

DEPARTMENT OF SUPPLY AND SERVICES

**STABILITY ENHANCEMENT OF COAL
MEASURES STRATA WITH AQUEOUS-BASED
CHEMICAL AGENT**

PROJECT NO. M1250



STABILITY ENHANCEMENT OF
COAL MEASURES STRATA WITH
AQUEOUS-BASED CHEMICAL AGENT

REPORT

BY

JACQUES, WHITFORD & ASSOCIATES LIMITED

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SUMMARY

This report describes the results of a preliminary effort to ascertain the effectiveness of surface applied chemicals as a swelling retardant for use in underground coal mines. Work on this project was conducted under the auspices of CANMET, contract serial number OSQ83-00152, Dr. Peter Cain Scientific Authority.

The three phases of the work reported are data collection, review and analysis and proposal for a laboratory test program.

Data was collected by discussion with the scientific authority, underground observations and a literature search. The detailed concept behind the research project was discussed and relevant data sources thoroughly explored. Through the courtesy of the Cape Breton Development Corporation, problem areas at the Lingan Colliery and the Prince Mine were observed first hand. Additional data relating to configuration of the workings, mining equipment operating practice and problems, water useage and chemistry, mineralogical studies and rock strengths were also procured.

The Lingan Colliery #3 W longwall is experiencing difficulties with chock penetration due to soft floor conditions. It is not obvious that this problem is related to softening of the floor strata due to water absorption because of limited duration of water exposure but rather to general low bearing capacity of the seat earth. There is potential for prolonged water exposure of the seat earth along the coal road and it is in this area where surficially applied chemical agents may be more effective.

At the Prince Mine softening and strength reduction of the seat earth due to water induced swelling of the strata was apparent. Reports of extreme operating problems relate to mine support structures, floor heave and general degradation of floor strata due to a combination of water absorption and mechanical working by mine equipment.



A literature search using four computerized data bases was conducted as well as a review of technical literature and data derived from commercial catalogues and other research organizations. A great deal of information is published in the area of swelling strata, however, the use of chemicals to stabilize strata within this context appears to be a unique concept.

The mechanisms of swelling are based on the physio-chemical behaviour of the clay minerals which comprise a substantial fraction of a typical seat earth composition. The subject of clay mineralogy, is developed and the processes governing the nature of the clay-water system have been described. Much of the observed swelling behaviour is directly related to the type or nature of the cations present in the pore fluid and adsorbed on the clay particles. It is this characteristic which provides the rationale for investigating chemical stabilizing agents.

Clay soil or clay shale structures also swell, or rebound, as a direct consequence of stress relief. The rate and degree of swelling are functions of in situ stress changes, as well as the availability of water: these factors are also part of the study of stability.

The oil well industry has actively used chemicals to stabilize shale formations, and practical methods of treatment which produce the required results are well established. Stabilization is promoted by the replacement of exchangeable cations by calcium, which is introduced in the form of lime or gypsum. Potassium hydroxide or potassium chloride were also determined to be effective. Recent developments include organic and inorganic polymers as stabilizing agents. Although the method of application is different for the subject of the report, the coal mining industry can benefit from the body of knowledge which has been developed by the petroleum industry.



Since the mining staff will be exposed to the shale stabilizers for extended periods of time; the health effects of the chemicals are of concern. According to information made available by distributors, most of the stabilizers are non-toxic, many being used as food additives. Material Safety Data Sheets are available and should be requested when purchasing a particular product.

Costs of the shale stabilizers are generally based on the quantities purchased, and therefore are negotiable.

In the context of the present terms of reference, work related to oil-well stabilization provides an excellent basis for the design of a laboratory test program. Prospects for such a program are presented in the report, based largely on this experience.

Although not clearly within our terms of reference, we have noted that underground conditions need clarification and that field studies should be done in consort with laboratory testing.



SOMMAIRE

Ce rapport a pour but de présenter les résultats obtenus lors d'une recherche préliminaire sur l'efficacité de l'application en surface de produits chimiques pour retarder le gonflement des planchers dans les mines de charbon souterraines. Les travaux de recherches ont été effectués sous les auspices du Centre Canadien de la Technologie des Minéraux et de l'Energie, contrat no. OSQ83-00152, dont M. Peter Cain, Ph.D. est le responsable scientifique.

Le rapport est présenté en trois parties:

- Informations et données recueillies
- Revision et analyse
- Programme proposé d'essais en laboratoire

Les données recueillies proviennent de discussions avec M. Peter Cain, Ph.D., de recherches bibliographiques, ainsi que d'observations faites dans des mines souterraines. Le cadre de la recherche a premièrement été discuté et par la suite les différentes sources d'informations disponibles ont été revisées à fond.

Avec la permission de la compagnie Cape Breton Development Corporation, les mines Lingan Colliery et Prince Mine ont été inspectées. Les problèmes mentionnés dans cette recherche ont été observés dans ces deux mines. D'autres données reliées à la configuration de la mine, à l'usage d'équipements miniers et des problèmes occasionnés, à l'utilisation d'eau et de ses propriétés chimiques, aux études minéralogiques et à la qualité du roc ont aussi été obtenues de la compagnie Cape Breton Development Corporation.

Un problème d'affaissement des supports installés pour soutenir le plafond existe dans le long mur #3 ouest de la mine Lingan Colliery. Ce n'est pas évident que la cause du ramollissement de la couche formant le plancher est dû à l'absorption d'eau, vu la courte durée d'exposition à l'eau, mais probablement dû à la faible capacité portante de l'assise. Par contre, le plancher du tunnel utilisé pour transporter le charbon est plus susceptible d'être exposé à l'eau et dans ce cas l'application, en surface, de produits chimiques peut être plus efficace.

Un ramollissement et une réduction de la capacité portante de la couche formant le plancher dû au gonflement causé par l'eau, étaient apparents à la mine Prince Mine. Des problèmes majeurs d'exploitation reliés aux structures de soutènement ainsi qu'au soulèvement et à la détérioration générale de la couche formant le plancher dû à une combinaison d'absorption d'eau et au remaniement causé par les équipements ont été notés.



Une recherche bibliographique à l'aide de quatre différents fichiers informatisés, ainsi qu'une revue de la littérature et des données techniques provenant de catalogues et autres organisations de recherches ont été effectuées. Il existe beaucoup de publications dans le domaine du gonflement des couches de terrains, cependant, l'utilisation de produits chimiques pour stabiliser ces couches de terrains, dans le contexte étudié, semble être unique en son genre.

Les mécanismes de gonflement sont basés sur le comportement physio-chimique des minéraux argileux qui constituent une fraction substantielle des couches sous-jacentes aux strates de charbon. Le domaine de la minéralogie des argiles et les processus gouvernant l'interaction argile-eau sont connus. La plupart des gonflements observés sont directement reliés au type ou à la nature des cations contenus dans l'eau interstitielle et l'eau absorbée liée aux particules d'argile. Cette caractéristique fournit le fondement pour effectuer des recherches sur l'utilisation de produits chimiques comme agents stabilisateurs. Les argiles et les argiles schisteuses gonflent aussi, ou rebondissent, après relâchement des contraintes. Le taux et le degré de gonflement sont fonctions des changements de contraintes ainsi que la disponibilité de l'eau. Ces facteurs font aussi partie de toutes les études de stabilité.

L'utilisation de produits chimiques pour stabiliser les parois de trous de forages dans les formations d'argile schisteuse est bien connue et développée par les compagnies pétrolières. La stabilisation se fait en remplaçant des cations échangeables par du calcium, qui est introduit sous forme de chaux ou de gypse. L'hydrate de potassium ou le chlorure de potassium sont aussi des agents stabilisateurs, de même que récemment des polymères organiques et inorganiques ont été trouvés efficaces. L'industrie minière peut profiter des connaissances développées par l'industrie pétrolière bien que la méthode d'application qui fait l'objet de ce rapport soit différente.

L'effet des produits chimiques sur la santé des employés est aussi un point à considérer car la plupart des mineurs y seront exposés pendant de longues périodes de temps. D'après l'information obtenue des fournisseurs locaux, la plupart des agents stabilisateurs sont non-toxiques et ils sont même utilisés comme additifs dans des produits de consommation. Une fiche donnant toutes les propriétés devrait être exigée pour garantir une utilisation sécuritaire du produit considéré.

Le coût des agents stabilisateurs est généralement basé sur la quantité achetée donc négociable.



Dans le cadre de cette recherche, les travaux reliés à la stabilisation des forages pétrolifères ont fourni une solide base pour établir un programme d'essais en laboratoire. Ce programme est présenté dans le rapport.

Quoique cet aspect ne fasse pas clairement partie de notre mandat, nous avons noté que les conditions souterraines devraient être précisées et qu'une étude sur le terrain devrait être effectuée de concert avec le programme d'essais en laboratoire.



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Title

1

Statement of Work

(x)



1. INTRODUCTION

1.1 Scope of Work

The strength and stability of underground coal measure strata is known to be affected by the absorption of water from various sources such as the mine atmosphere, water sprays used for dust laying and also from connate water. One of the effects frequently associated with water absorption is strength reduction due to the presence of swelling clays within the mine shales. The Cape Breton Coal Mine Research Laboratory (CBCMRL) commissioned Jacques, Whitford and Associates Limited (JWA) to conduct a preliminary review of the problem, review the range of oil well chemicals available and to propose a laboratory test program to evaluate the practicality and efficiency of some of the chemicals for use in underground coal mines. The terms of reference of the assignment are attached as Appendix 1. Approximately 30 professional man days of effort were budgeted for the completion of the entire task.

1.2 Conduct of the Study

Immediately following the awarding of the contract in November 1983, arrangements were made to meet with Dr. Peter Cain, Scientific Authority, and to tour the two problem areas underground at mines operated by the Cape Breton Development Corporation (CBDC).

The purpose of the underground visit was to acquaint the geotechnical engineering personnel with the physical arrangement underground and the operational aspects of the mine. The two locations examined, were at the Lingan colliery New Waterford and also at the Prince Mine, Pt. Aconi.



A literature search was conducted to ascertain the nature and results of any prior published work using company files, Canmet Data and library facilities available in Halifax. Inquiries were also made with the USBM in Pittsburgh, Pa. and at SMRE in Sheffield, U.K. Additional data was also secured from Haliburton Services and Dr. N. Morgenstern as well as mud company literature and catalogues.

This report is presented in seven sections. Sections 1, 2, and 3 deal with the conduct of the study task, the underground site examination and the literature search respectively. Section 4 briefly reviews pertinent technical aspects of swelling strata. Section 5 presents a discussion of chemicals with potential for use as stabilizing agents. Section 6 develops a recommended laboratory test program for future work followed by general recommendations in Section 7.

The work reported herein was conducted by the following JWA personnel.

Dr. J.D. Brown, P.Eng.	- Senior Geotechnical engineer
Dr. B. Taylor	- Geotechnical Engineer
Mr. J.A. Amirault, P.Eng.	- Mining Engineer

The writers gratefully acknowledge the participation of various persons who assisted in the assembly of data and the preparation of this report. We particularly want to thank Dr. Peter Cain, Scientific Authority, CBCMRL, Mr. Steve Forgeron, CBDC, Dr. N. Morgenstern and personnel from Haliburton.



2. EXAMINATION OF UNDERGROUND WORKINGS

The CBDC operate three collieries; #26, Langan, Prince and are developing a fourth at Donkin, within the Sydney coal basin located on Cape Breton Island (see Fig. 1). The underground mine examination was conducted at the Langan and Prince collieries which are known to exhibit particular problems with soft floor conditions.

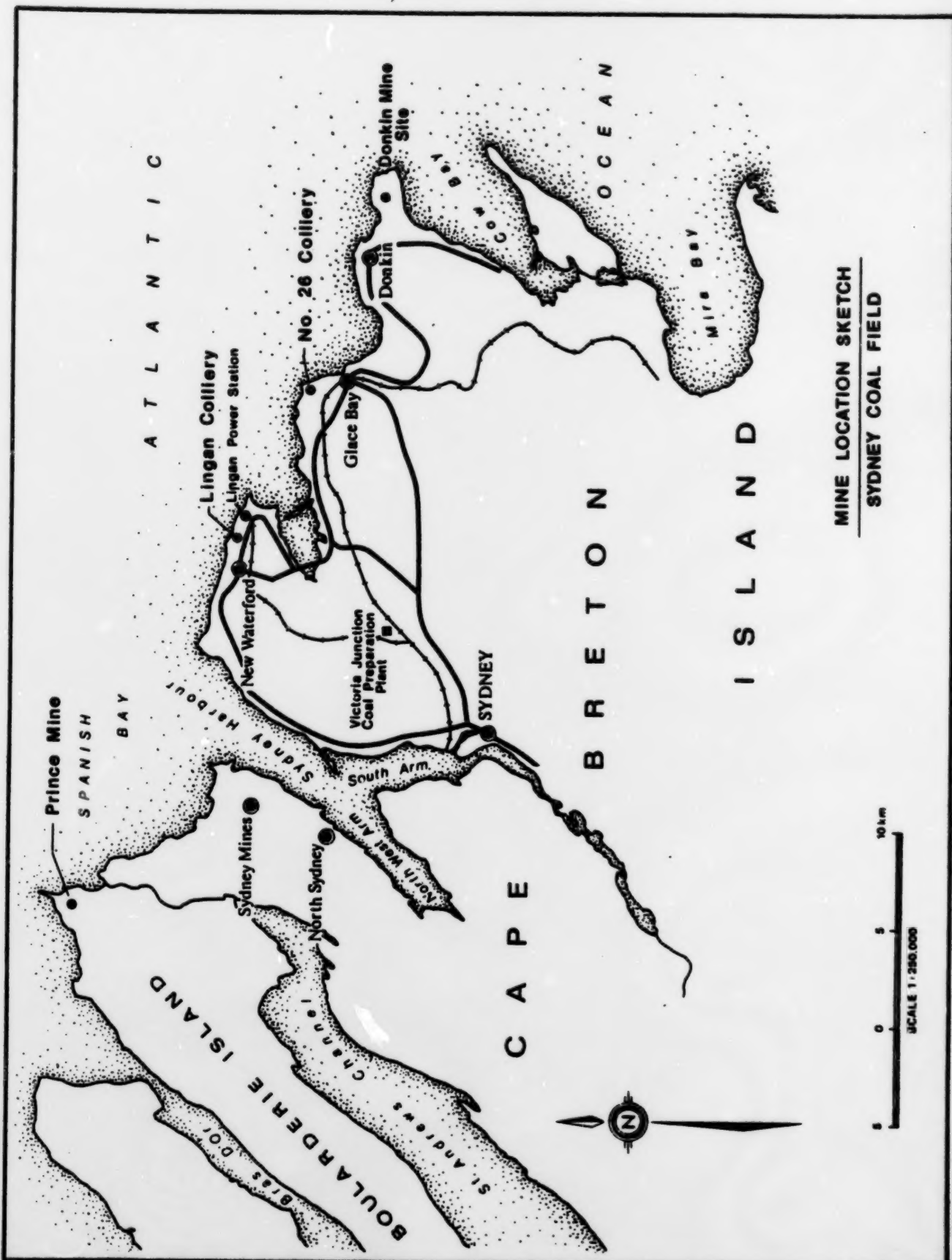
2.1 Langan Colliery

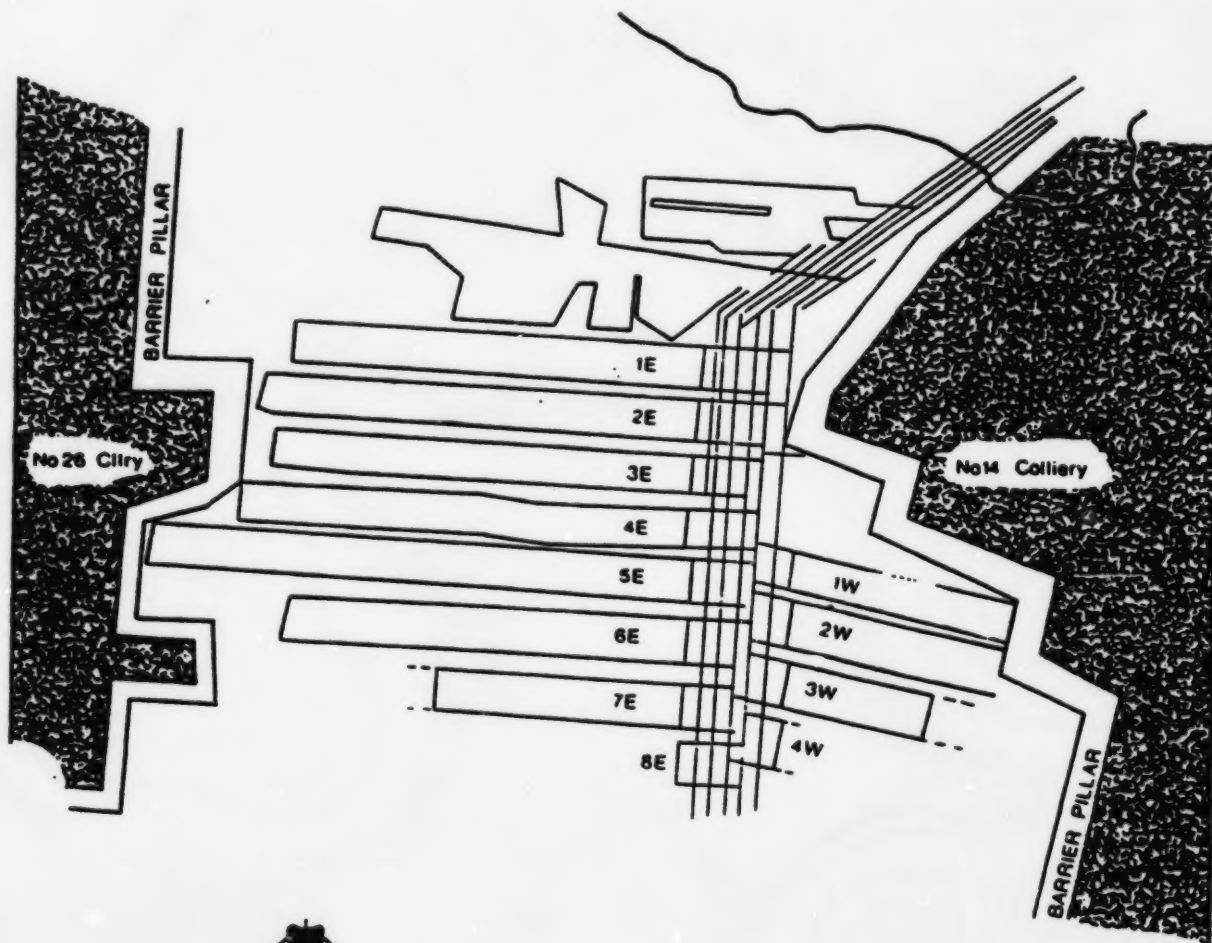
The Langan Colliery operates on the Harbour seam which in this area is approximately 2 metres thick. The underlying seat earth is in excess of one half metre in thickness and contains over 50% clay minerals. The seat earth (mudstone) exhibits a low compressive strength 24 MPa*.

The Langan colliery produces approximately 1.4 million tonnes per annum from underground longwall mining operations. Longwall faces are approximately 200 metres in length and are separated from adjacent walls by a 60 metre coal pillar. At the time of examination, #3W wall had been operating for 2 years, approximately 1,500 metres from the main slopes and was nearing completion of its productive life. The general arrangement of the walls at Langan colliery are shown in Figure 2.

The coal is won using an Anderson Strathclyde double drum ranging shearer to cut the coal which is discharged to an Armoured Face Conveyor (AFC) and to subsequent conveyors to be hoisted from the mine. The geometry and dimensions of the mining equipment require an opening of approximately 3 metres x 2 metres to be maintained.







LONGWALL PANEL ARRANGEMENT
LINGAN COLLIERY

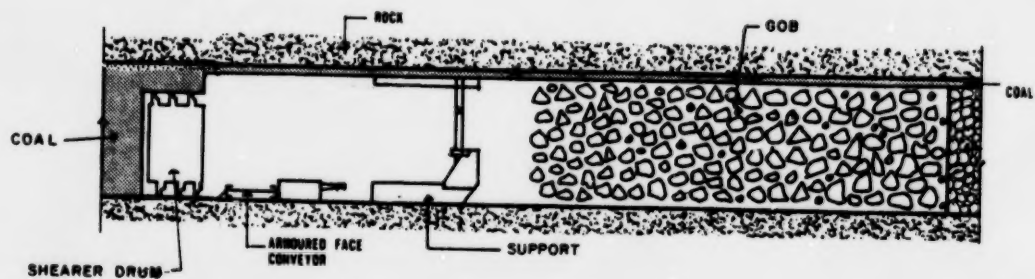
(after EMR)

Wall face support is provided by 2 legged hydraulic Hemscheidt shield mine supports (chocks) positioned at 1.5 metres center to center with a capacity of 250 tonnes each. Normal operation requires that the supports are advanced sequentially in the direction of mining to control the newly exposed roof as the shearer passes. The supports are depressurized, advanced and repressurized in the new position. The general arrangement of the 2 legged supports is depicted in Figure 3. Severe mining induced stresses due to the removal of the coal and the cyclic loading pressures of the support chocks serve to disturb and "work" the underlying seat earth.

The #3W wall had been experiencing problems with chock penetration into the floor strata-seat earth. The problem was most evident near the center of the wall face where floor penetration was in excess of 45 cm which obviously caused severe operating and economic problems to the mine operator.

The mine operators had attempted to decrease the bearing pressure of the chocks through the addition of skid plates which were not totally effective. It was not clear to the writer that this soft floor condition was due to softening of the strata due to water but rather to a problem relating more to the natural bearing capacity of the material. Samples taken for examination in the laboratory showed low strength characteristics. When subjected to soaking in water there was no tendency to degrade further. The material did reveal marked slickensides due to pressure effects. These slickensides are believed to have been present prior to chock loading.





FACE SUPPORTS
LINGAN MINE

Water use for dust laying purposes is reported by the mine operator to be in the order of 150 to 190 litres/min on the Anderson Shearer used on the longwalls. It is probable that the majority of this water is taken up by the fine coal while mining is in progress. During the frequent halts in production, the water continues to flow to a lesser extent and does afford the potential to wet the underlying seat earth. Any water will quickly flow down the dip of the coal strata to the coal haulage level.

A more severe exposure of the seat earth to water occurs along the coal road where water frequently pools in low areas thus permitting extended wetting periods. In these cases, strata with the propensity to swell has ample opportunity for exposure to excess water. This is a particularly critical condition because the coal haulage level, unlike the wall face itself, must be maintained open for the life of the wall face (24-36 mos) and is also subjected to increasing stress due to mining. Floor heave due to whatever cause - stress related or swelling strata, is most frequently observed on the coal haulage levels and it is here that chemical treatment may be most effective.

Analysis of mine water from a wall face provided by CBDC showed the following characteristics:

Ph	- 6.96
Conductivity	- 20,000 + mhos/cm
Chlorides	- 4,950 mg/l



2.2 Prince Mine

The Prince Mine is located at Pt. Aconi and has been operating on the Hub Seam since 1975. (Figure 4) Seam height is variable with a clean upper leaf of 1.4 to 2 metres underlain by one half metre of "inferior bench coal". The seam dips seaward, to the north, at 6 degrees.

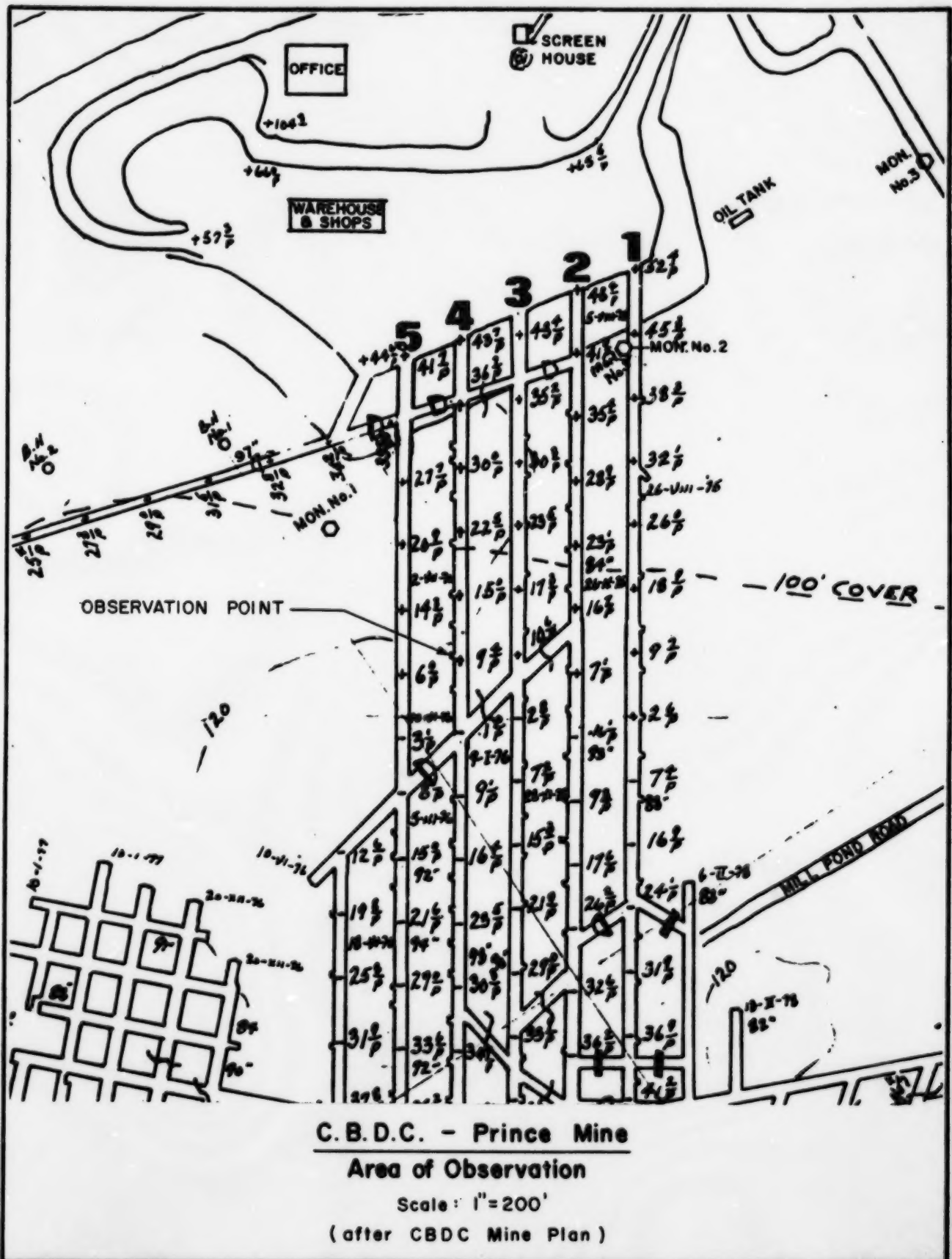
Initial attempts to mine the Hub Seam were by room and pillar methods using mobile equipment. In addition to other operating difficulties, several problems were encountered with soft floor conditions. In recent years a "shortwall" system has been adopted and is successfully reporting record productivity levels. Following poor production performance during the years 1975 to 1979 marked improvement has been demonstrated. In 1983, the colliery reported production of 735,000 tonnes of raw coal.

Observations at the Prince Mine were made several hundred feet inbye the portal on the material slope. (Figure 5) The seat earth was exposed in a manhole over an area of approximately .4 to .6 sq. metres. The material is reported by the geologist to be fairly extensive throughout the mine and is in the order of 1 metre in thickness.

Pocket penetrometer tests on the material showed variable low strengths. The material was visibly wet and plastic. The mine operator informs that when freshly exposed, the material is dense and exhibits strength. When exposed to the unusually wet conditions in this mine the seat earth quickly degrades in strength to the material that was observed.

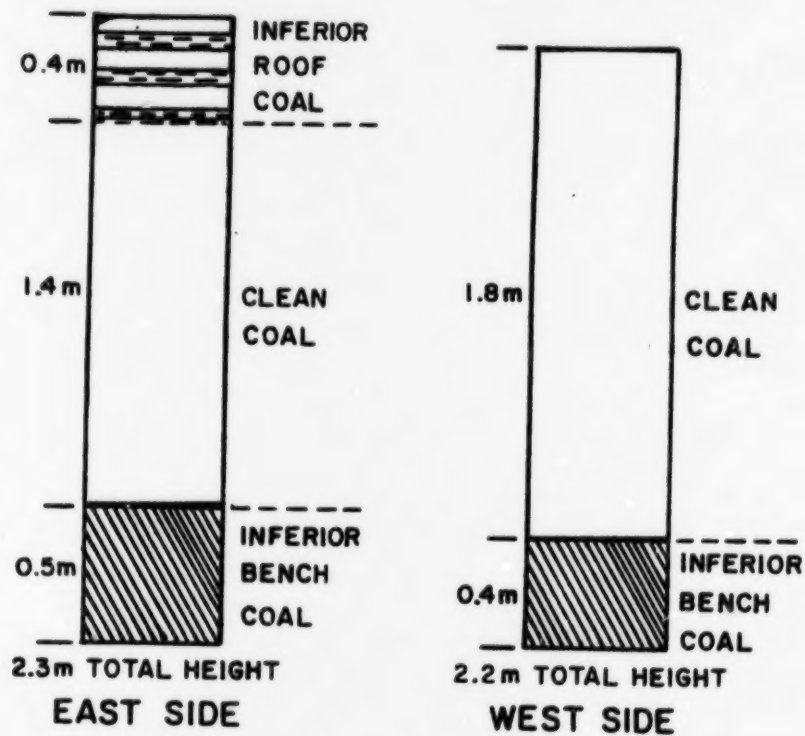
Exposure of the seat earth to water and subsequent softening has been the cause of several operational problems. Within the slope ground support is provided by transverse H beams set on steel posts. The posts have been observed punching the seat earth as the strength reduces. The seat earth has also been observed to extrude from beneath the coal rib into the mine opening resulting in floor heave and related problems of closure, track misalignment and rib pillar failure. During the early stages of the mine, rubber tired shuttle cars were used for coal transport. Severe operating problems developed as the seat earth degraded to "mud".





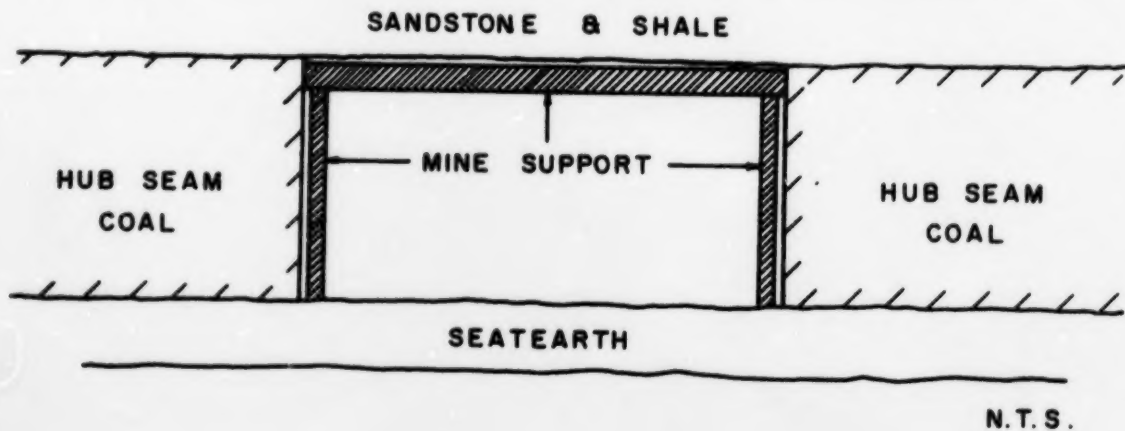
PRINCE MINE - HUB SEAM

TYPICAL SEAM SECTIONS



(after C.B.D.C. Mine Plan)

SLOPE SUPPORT ARRANGEMENT



The current shortwall operations, in use at the Prince colliery, were not observed nor were any comments made relative to soft floor conditions. The writers are informed that in general the operating problems do not appear to be as severe as in the past. This may be due, in part, to the increase in operating depth and therefore increased burden pressure on the strata and also the tendency towards decreased permeability and water flow through the strata. These effects would serve to limit the flow of surface water into the mine openings at the location of current mining operations and hence reduce water related problems. The effects of water derived from water sprays, will remain as described at the Langan Colliery.



3. LITERATURE SEARCH

In order to assess the information available in the literature, a number of computer data bases were accessed. Table 1 presents a list of data bases, the keywords used in each search, the number of keyword "hits", and the numbers of titles, abstracts and copies of text obtained. Titles to be obtained were selected on the basis of those references containing only appropriate combinations of selected keywords. Abstracts and text to be obtained were determined after a review of the titles.

We are directed to the NCB through contact with SMRE. Unfortunately, time was not available to pursue the NCB. Attempts should be made in the future to ascertain the results of any relevant work known to the NCB.

Correspondence with the USBM suggested several companies which were using resins and coatings largely related to ground support. We therefore did not pursue these avenues further.



Table 1

LITERATURE SEARCH SUMMARY

Date Base	Key Words	Keyword Hits	Titles		Abstracts		Text	
			Obtained	Obtained	Obtained	Obtained	Obtained	Obtained
APILIT	stabil*, swelling, borehole, well, clay, shale, chemical	116,209	204		204			
MINTEC	coal, floor heave, swell*		20		20			
EI(COMPENDEX)	coal, measures, stabil*, swelling, clay, pressure, floor, heave, mine, chemical	138,909	14		3			5
NCB (COAL ABSTRACTS)	swelling, clay, pressure, coal, min*, soil, stabiliz*, floor, heave, chemical, strata, measure*	94,293	53		9			

4. SWELLING CLAYS

4.1 General

The purpose of this section is to pinpoint the reasons for the softening of seat earths. Both oil field engineering and civil engineering have to deal with problems which display some of the same characteristics, and much effort has been put into studies of cause, effect, and remedies. It is worthwhile, therefore, to review some basic concepts which can provide insights into the behavior of seat earth.

The characteristic common to mining, civil, and oil well experience is that there exists a class of materials which swell and soften when subjected to stress relief, in the presence of water. These materials exist in geological formations which may be of considerable age - at least as old as the upper Paleozoic - and which in normal parlance would be called "rock". They may also be much younger, but it is with the older "rocks" that we are most concerned. They are almost universally referred to as shales, but this name presents some concern in that wide differences of opinion exist in regard to the classification and identification of shales.

Underwood (1967) provides a valuable starting point in order to understand this class of materials. His classification of sedimentary rocks (Fig. 6) distinguishes between "soil-like or compacted" shale and "rock-like or cemented" shale.¹ In the use of these terms he follows Mead (1936). This distinction is broken down further in the shale classification given in Figure 7. In this classification the soil-like shales include clay shale, silty shale, sandy shale and black shale. The clay shales have received much attention because they are notorious for creating difficulties in geotechnical engineering (Morgenstern 1979), and may also be the most important in the present context.

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¹ Terzaghi (1964) described two simple properties which can be used to distinguish rock-like from soil-like shales: 1) they should produce a clear ring when struck by a hammer, and 2) they should show no volume change when immersed in water.



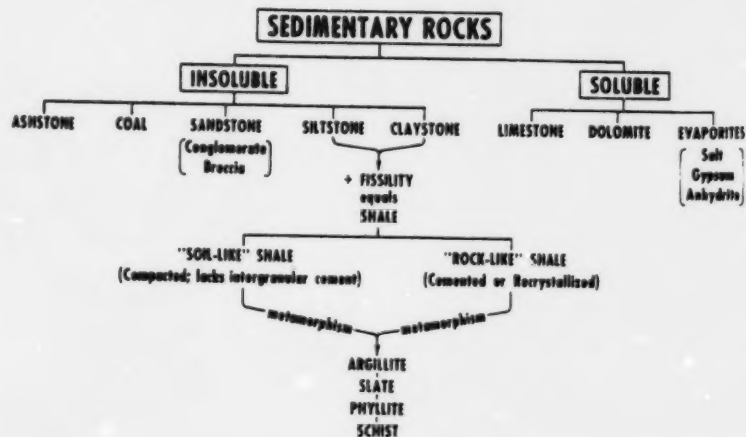
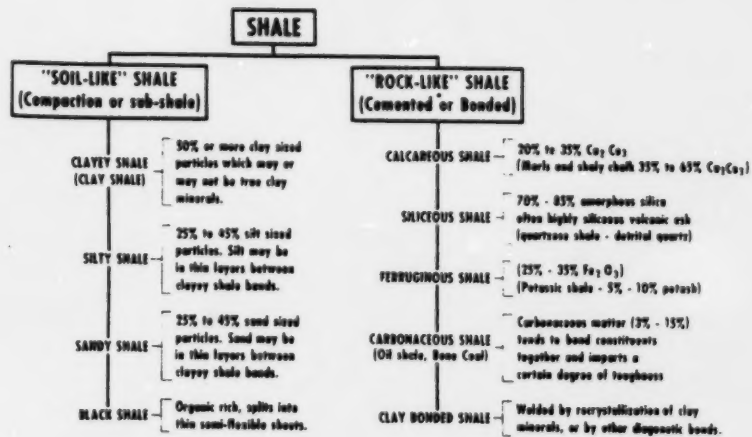


FIG. 6.—CLASSIFICATION OF SEDIMENTARY ROCKS



*The cemented shales can be further classified according to grain size, i.e., sandy, silty, clayey.

FIG. 7 —A GEOLOGICAL CLASSIFICATION OF SHALE

after Underwood (1970)

Underwood describes a number of the significant physical properties of shale and attempts to relate them to probable in situ behavior (Table 2).

Morgenstern and Eigenbrod (1974) concentrated on the softening-upon-immersion in water aspect as an aid to classifying argillaceous soils and rocks. This approach was selected because it is the water deterioration characteristics which are important for many practical applications. Among these they include durability of tunnel walls, which returns us to the question of seatearths.

It seems a constructive hypothesis to suggest that coal measures shales can be included in the shale classifications cited above, and that the potentially troublesome seat earths can be placed in the "compaction shale" category. Furthermore, the troublesome materials exhibit the characteristics which in the literature fall under the collective heading "swelling clays". This hypothesis is presented in Figure 8.

4.2 Swelling Processes

The reasons for swelling, and associated softening and slaking, can be found in the mineralogy and chemistry of the clay particles. Non-clay minerals are also present in all clay shales and clays, but they are essentially inert. Compaction shales and soft clays are basically alike in the characteristics and fundamental behaviour of their clay minerals. Hence a good starting point in the present study is a brief review of clay mineralogy, and what may be called the "chemical" aspect of swelling behaviour.

The propensity of a clay mass to change volume is also dependent on the externally applied stresses. In particular reference to swelling, the question is that of stress relief. This can be thought of as the "mechanical" aspect of swelling behaviour.

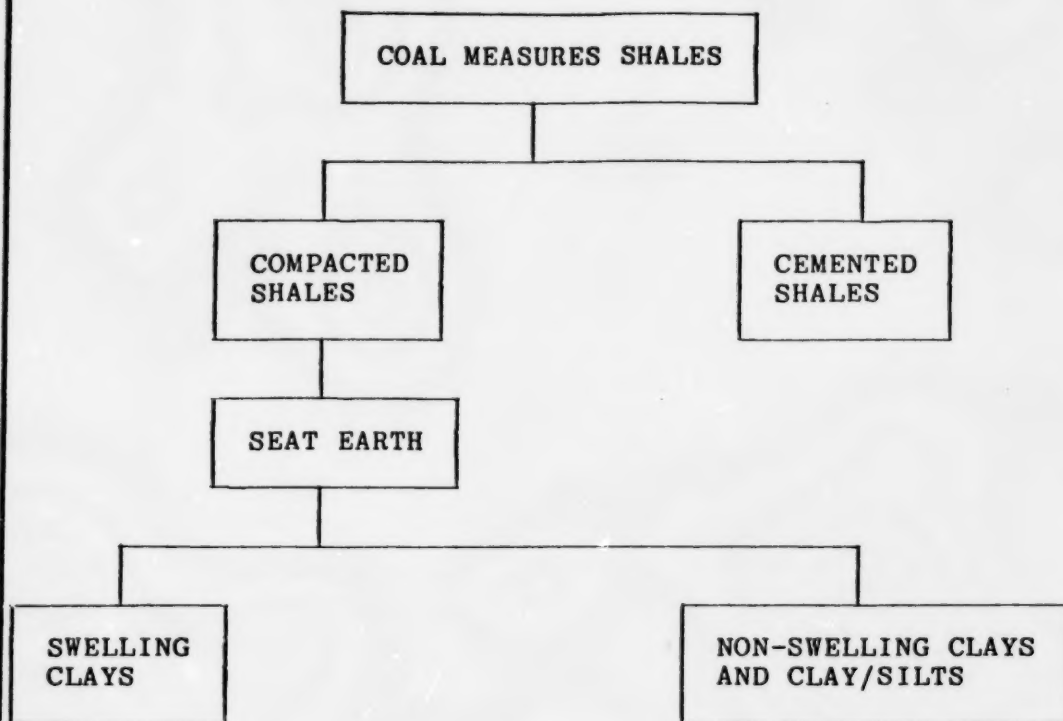
In this Section the chemical and mechanical aspects of the swelling of clays will be reviewed briefly.



TABLE 2.—AN ENGINEERING EVALUATION OF SHALES

Physical Properties			Probable <u>in-situ</u> Behavior						
Laboratory tests and in-situ observations	Average range of values		High pore pressure	Low bearing capacity	Tendency to rebound	Slope stability problems	Rapid slaking	Rapid erosion	Tunnel support problems
(1)	Unfavorable (2)	Favorable (3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)
Compressive strength, in pounds per square inch	50 to 300	300-5000	✓	✓	---	---	---	---	---
Modulus of elasticity, in pounds per square inch	20,000 to 200,000	200,000 to 2×10^6	---	✓	---	---	---	---	✓
Cohesive strength, in pounds per square inch	5 to 100	100 to >1500	---	---	✓	✓	---	---	✓
Angle of internal friction, in degrees	10 to 20	20 to 65	---	---	✓	✓	---	---	✓
Dry density, in pounds per cubic foot	70 to 110	110 to 160	✓	---	---	---	---	✓(?)	---
Potential swell, in percentage	3 to 15	1 to 3	---	---	✓	✓	---	✓	✓
Natural Moisture content, in percentage	20 to 35	5-15	✓	---	---	✓	---	---	---
Coefficient of permeability, in centimeters per second	10^{-8} to 10^{-10}	$>10^{-8}$	✓	---	---	✓	✓	---	---
Predominant clay minerals	Montmorillonite or Illite	Kaolinite & Chlorite	✓	---	---	✓	---	---	---
Activity ratio - Plasticity Index Clay content	0.75 to >2.0	0.35 to 0.75	---	---	---	✓	---	---	---
Wetting and drying cycles	Reduces to grain sizes	Reduces to Flakes	---	---	---	---	✓	✓	---
Spacing of rock defects	Closely Spaced	Widely Spaced	---	✓	---	✓	---	✓(?)	✓
Orientation of rock defects	Adversely Oriented	Favorably Oriented	---	✓	---	✓	---	---	✓
State of stress	>Existing Over-burden Load	✓ Over-burden Load	---	---	✓	✓	---	---	✓

after Underwood (1970)



A SUGGESTED CLASSIFICATION OF COAL MEASURES SHALES

4.2.1 Clay Mineralogy

Clay minerals are composed of sheets of molecules, with the sheets bound to each other by atomic bonds of varying strengths. The sheets are made up of units, as shown in Figure 9, which contain silicon, aluminum, magnesium, etc. as positively charged atoms.

When the "right" atom is in place, the sheets or combinations of sheets ("layers") are electrically neutral. However, due to isomorphous substitution of these atoms by others of different electrical charge, the sheets or layers invariably have a net positive charge deficiency, which is balanced by adsorbed, exchangeable cations held on the outside of the sheets. The nature of the isomorphous substitution and the resultant charge deficiency explain most of the differences between the common clay minerals. The nature of the adsorbed cation explains much of the physical behavior of the clay particles made up of bundles of these sheets and layers. The structures of some of the common clay minerals are shown in Figures 10 and 11.

The permanent net negative charge carried by the clay particle is measured by the cation exchange capacity.

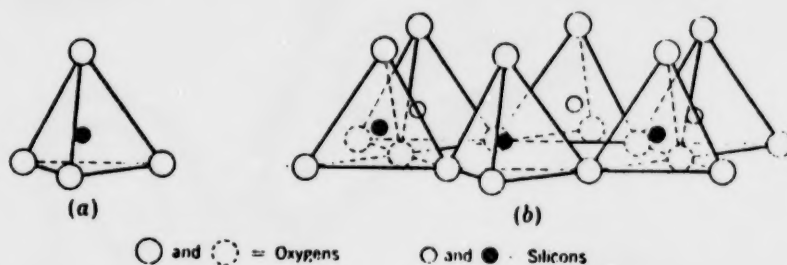
$$\text{CEC} = \frac{\text{electrical charge deficiency}}{\text{unit mass}}$$

usually expressed as milliequivalents of electric charge per 100 grams of particle weight. The CEC is useful in assessing the potential for swell, as well as plasticity and other characteristics.

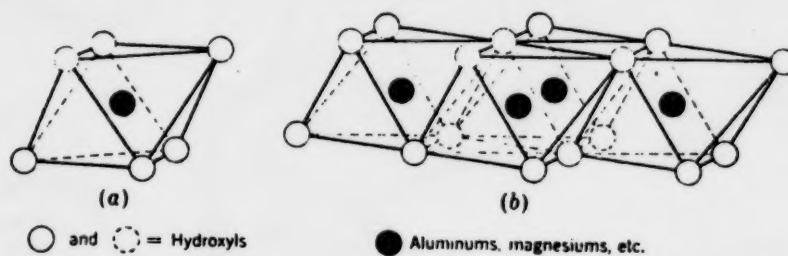
The exchangeable cations are positively charged particles. In a clay suspension in water, or in a saturated clay soil, the cations and water molecules are free to move in the fluid phase, but they take up positions adsorbed on the particle faces, for the reasons given above. Therefore, when a clay particle is dropped into water, both the mineral surfaces and attached exchangeable ions pick up water i.e. hydrate¹.

¹ compare with oil-well definition of hydration, Sect.4.2.2



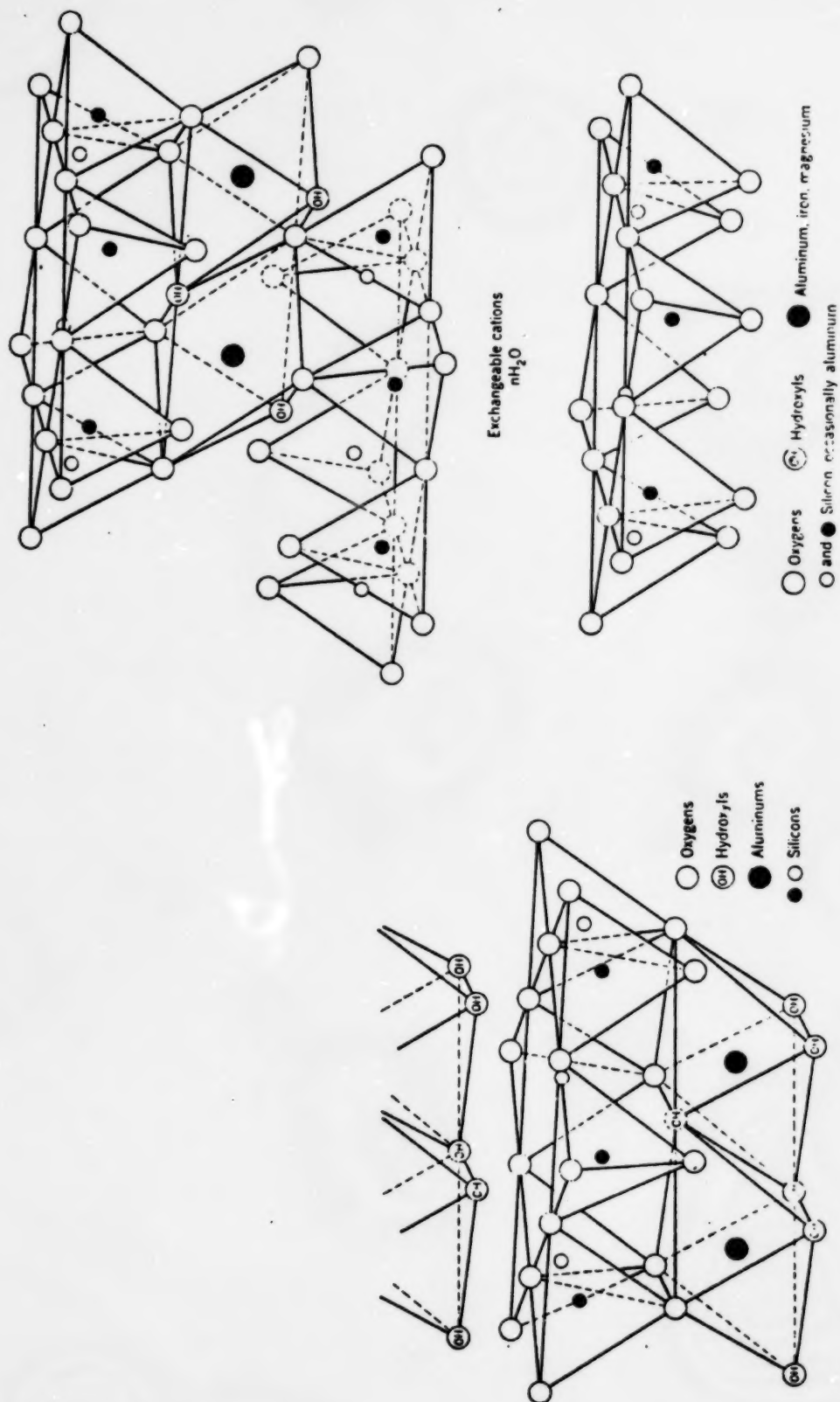


(a) single silica tetrahedron and (b) the sheet structure of silica tetrahedrons arranged in an hexagonal network



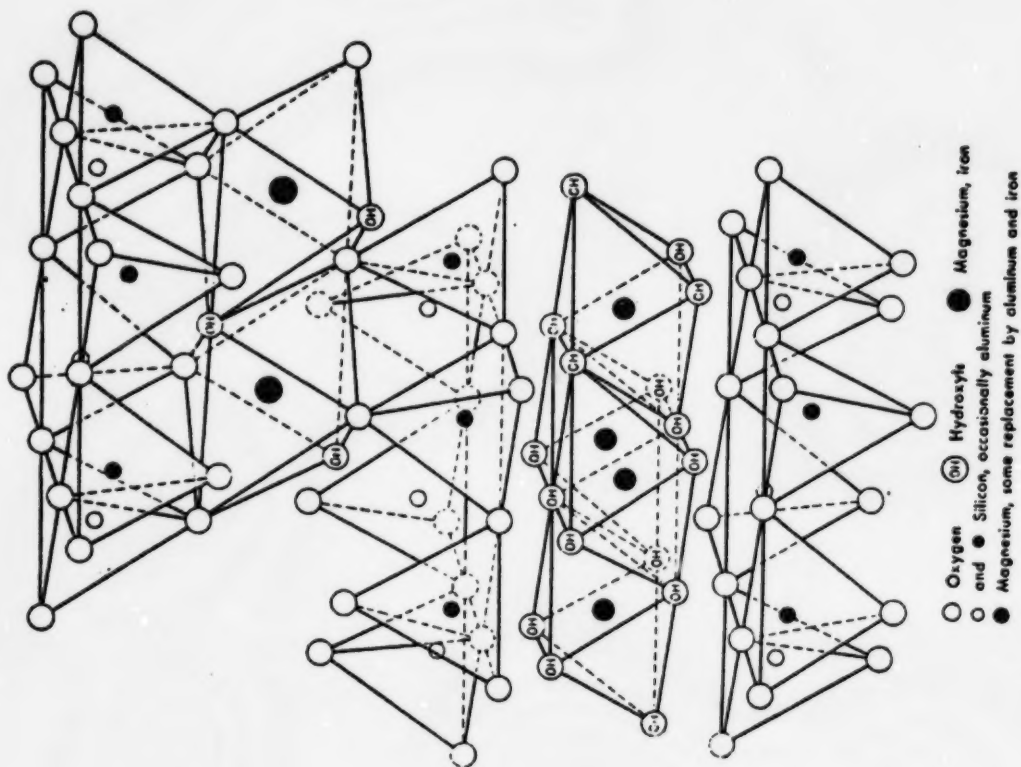
(a) single octahedral unit and (b) the sheet structure of the octahedral units

CLAY STRUCTURE

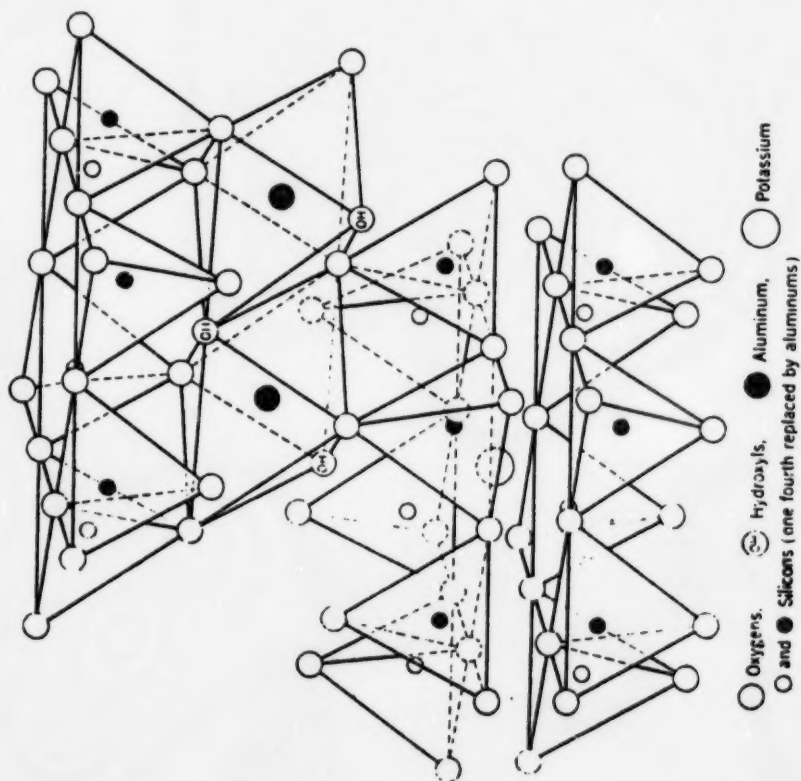


Kaolinite

Montmorillonite



Chlorite



Muscovite

CLAY MINERALS

The ions with their shells of water move away from the mineral surface to positions of equilibrium, to produce the "double layer". This behavior is at the root of swelling characteristics since different ions result in different double layer thicknesses, which in turn change the properties of the mass of the particles. There are general principles which control the rate and direction of the exchange reactions. These principles involve valence and concentration of cations, pH, etc.

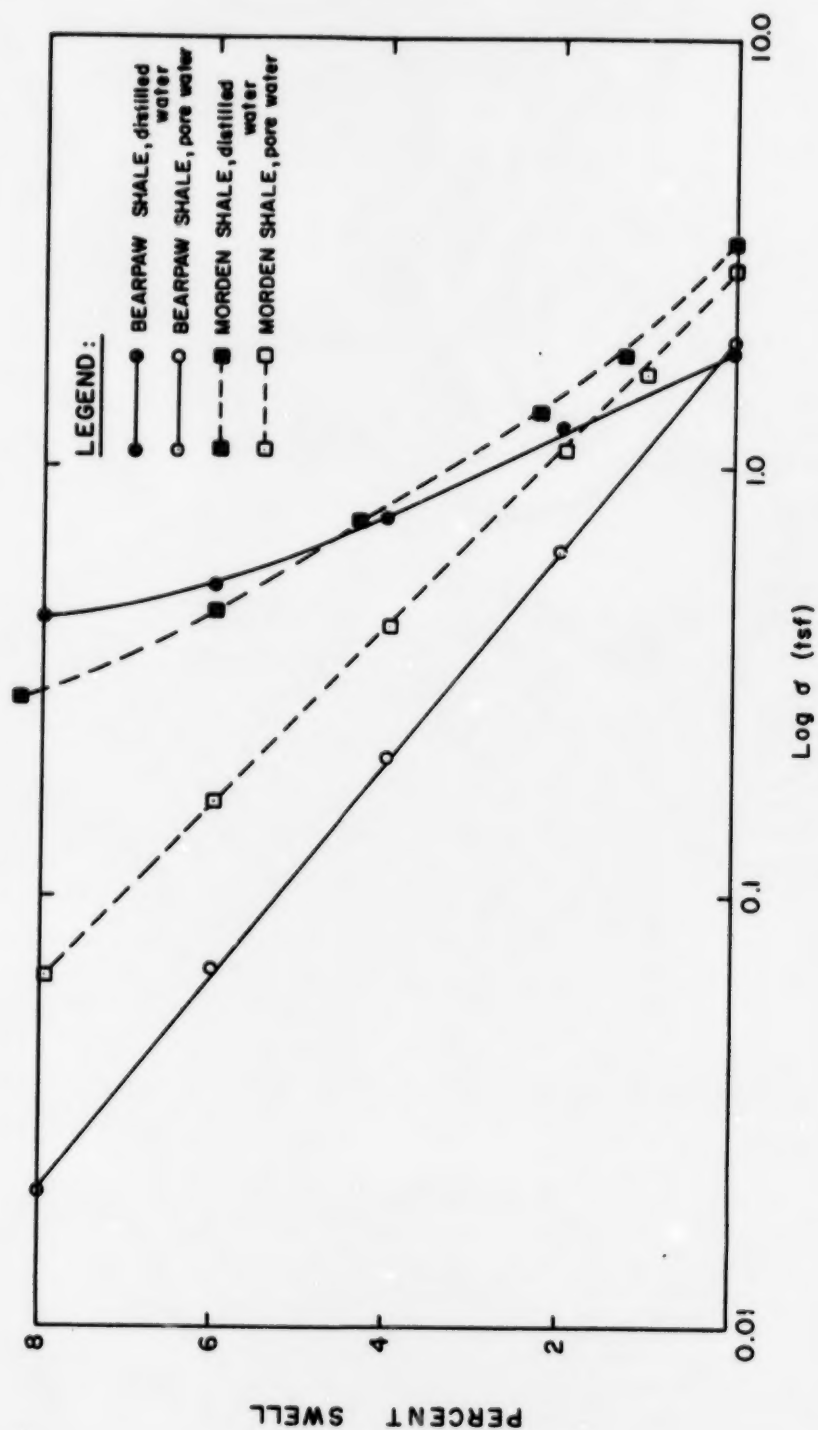
4.2.2 Chemical and Mechanical Aspects of Swelling

The swelling process of clays and clay shales in situ is broken into two phases by O'Brien and Chenevert (1973): a) uptake of water due to differences between the electrolyte components of the shale pore water and the external fluid. b) uptake of water due to stress release. They note that the former process has much greater swelling potential.

According to Peters and Stout (1977) the clay particles are in flocculated equilibrium with their native fluids. Reduction of cation concentration by dilution with induced fluids such as mine water can occur, and will result in dispersion. They postulate that a phenomenon similar to osmosis exists, and the weaker solution forces its way into the clay lattice in an attempt to dilute the dense atmosphere of cations surrounding the clay plates. In this way the clay takes up the fresh water.

A more definitive description is given by Morgenstern and Balasubramanian (1980). Two shales, the montmorilloritic Bearpaw shale and the predominantly illitic Morden shale were allowed to swell both in their connate pore fluids and in distilled water, after first establishing the swelling pressure for which the volume change was zero. The results presented in Figure 12 show the controlled swell in the pore fluid, which was due to stress release only. They also show the controlled swell in distilled water, which was significantly greater because of the differences in salt concentrations. The authors were able to demonstrate that





SWELLING IN TWO WESTERN CANADIAN SHALES

the swelling in distilled water is predictable on the basis of double layer theory, and that the double layer repulsion can be expressed as a component of the effective stress, i.e.

$$\sigma' = \sigma - u - (R-A)$$

where σ' = effective stress

σ = total stress

u = pore water pressure

$(R-A)$ = component of effective stress associated with physico-chemical changes

Engineering experience and the study of clay mineralogy have identified certain clay minerals which are more susceptible to swelling than others, although the actual amount of swelling depends upon other factors such as the exchangeable cations, pH, etc. Thus, the clay minerals can be arranged in descending order of swelling potential as follows:

sodium montmorillonite
calcium montmorillonite
illite (hydrous mica)
kaolinite

Tables 3 and 4 provide an illustration of the relative magnitudes of swelling of some clays and electrolytes as reported by different sources.

The most common cations occurring naturally are Na^{++} , Ca^{++} , Mg^{++} , and K^{+} . Of these, sodium has been found to have an important bearing on the structural status of a clay, and is often described in terms of the exchangeable sodium percentage (ESP) where

$$\text{ESP} = \frac{\text{Na}^{+}}{\text{CEC}} \times 100$$

For a more comprehensive treatment of this topic than this very brief outline provides, a number of standard reference texts can be consulted (e.g. Van Olphen 1963, Mitchell 1975, Chen 1975).



Table 3 Swelling Index Values for Several Minerals

Mineral (1)	Pore Fluid, Adsorbed Cations, Electrolyte Concentration, in Gram Equivalent Weights per Liter (2)	Void Ratio at Effective Consolidation Pressure of 100 psf (3)	Swelling Index (4)
Kaolinite	Water, sodium, 1	0.95	0.08
	Water, sodium, 1×10^{-4}	1.05	0.08
	Water, calcium, 1	0.94	0.07
	Water, calcium, 1×10^{-4}	0.98	0.07
	Ethyl alcohol	1.10	0.06
	Carbon tetrachloride	1.10	0.05
	Dry air	1.36	0.04
Illite	Water, sodium, 1	1.77	0.37
	Water, sodium, 1×10^{-3}	2.50	0.65
	Water, calcium, 1	1.51	0.28
	Water, calcium, 1×10^{-3}	1.59	0.31
	Ethyl alcohol	1.48	0.19
	Carbon tetrachloride	1.14	0.04
	Dry air	1.46	0.04
Smectite	Water, sodium, 1×10^{-1}	5.40	1.53
	Water, sodium, 5×10^{-4}	11.15	3.60
	Water, calcium, 1	1.84	0.26
	Water, calcium, 1×10^{-3}	2.18	0.34
	Ethyl alcohol	1.49	0.10
	Carbon tetrachloride	1.21	0.03
Muscovite	Water	2.19	0.42
	Carbon tetrachloride	1.98	0.35
	Dry air	2.29	0.41
Sand			0.01 to 0.03

From Olson and Mesri (1970).

Swelling or Compression Index =

$$\frac{e_1 - e_0}{\log_{10} p_1 - \log_{10} p_0}$$

where e = void ratio
 p = consolidation pressure

after Mitchell (1976)

Table 4 COMPRESSION AND SWELL OF CLAY MINERALS^a

Clay	Exchangeable Ion	w _i	w _p	Compression Index	Swell ^b Index
Montmorillonite	Na ⁺	710	54	2.6	—
	K ⁺	660	98	1.0	—
	Ca ²⁺	510	81	2.2	0.51
	H ⁺	440	55	1.9	0.34
	Mg ²⁺	410	60	1.9	0.44
	Fe ³⁺	290	75	1.6	0.03
	Fe ³⁺ dried and rewet	210	63	1.7	0.006
Illite	Na ⁺	120	53	1.10	0.15
	K ⁺	120	60	0.62	0.27
	Ca ²⁺	100	45	0.86	0.21
	H ⁺	100	51	0.61	0.10
	Mg ²⁺	94	46	0.56	0.18
	Fe ³⁺	110	49	—	0.15
	Fe ³⁺ dried and rewet	100	46	0.50	0.22
Kaolinite	Na ⁺	53	32	0.26	—
	K ⁺	49	29	—	0.06
	Ca ²⁺	38	27	0.21	0.06
	H ⁺	53	25	0.23	0.05
	Mg ²⁺	54	31	0.24	0.08
	Fe ³⁺	59	37	0.24	0.06
	Fe ³⁺ dried and rewet	52	35	0.19	0.15
Attapulgite	Mg ²⁺	270	150	0.77	0.24

COMPRESSION AND SWELL OF CLAY MINERALS^c

Clay	Exchangeable Ion	w _i	Compression Index	Swell ^d Index
Montmorillonite	Li	576	7.7	2.0
	Na ⁺	494	6.2	2.5
	K	193	2.0	0.3
	Ca ²⁺	186	2.0	0.8
	Ba ²⁺	168	1.4	0.2
Kaolinite	Na ⁺	98	0.6	0.2

RECOMPRESSION AND SWELL OF CLAY MINERALS^e

Mineral	Exchangeable Ion	Pore Fluid	Compression Index		Swell Index	
			1000 to 100 kN/m ²	100 to 10 kN/m ²	1000 to 100 kN/m ²	100 to 10 kN/m ²
Montmorillonite	Na ⁺	10 ⁻³ M NaCl	9.0	17.0	9.0	17.0
	Ca ²⁺	10 ⁻³ M CaCl ₂	5.0	9.0	—	—
Illite	Na ⁺	0.001 to 0.1 M NaCl	0.9	1.7	—	—

^a From Cornell, 1951.

^b Estimated from Cornell data.

^c From Jimenez Salas and Serratosa, 1953.

^d Estimated from the Jimenez Salas and Serratosa data.

^e From Bolt, 1956.

after Lambe and Whitman, 1969

In the compaction shales, the high density and the moderately high strength and stiffness which are associated with the unswelled state are attributed to the consolidating effect of many metres of overburden, which may have been thicker in the past than it is today, and also to the consolidating effect of dessication processes. In the first of these processes, the mineral grains have been squeezed together. In this squeezing process, the volume decreases by three mechanisms; i) sliding of grains past each other to produce a denser state of packing; ii) elastic deformation including bending of platy particles; and iii) compression of the double layers at clay contact points. The high pore water tensions associated with desiccation also force the particles close together. An additional effect of dessication is to drive off some of the water of hydration.

When these shales are subsequently unloaded, either by overburden removal or by the stress relief effects of a nearby excavation, borehole, tunnel, etc., and water is present, the above mechanisms tend to reverse, and the volume increases.

A typical volume change versus consolidation pressure curve is shown in Fig. 13. The slope of the unloading curve defines the swelling index, C_s . Typical values of C_s are given in Tables 3 to 5.

A study of swelling due to unloading can provide insight into the potential magnitude of volume change, and concurrent change in strength. In addition, an understanding of in situ behaviour requires a knowledge of the rate at which swelling occurs since swelling is a time-dependent process as illustrated in Fig. 13.



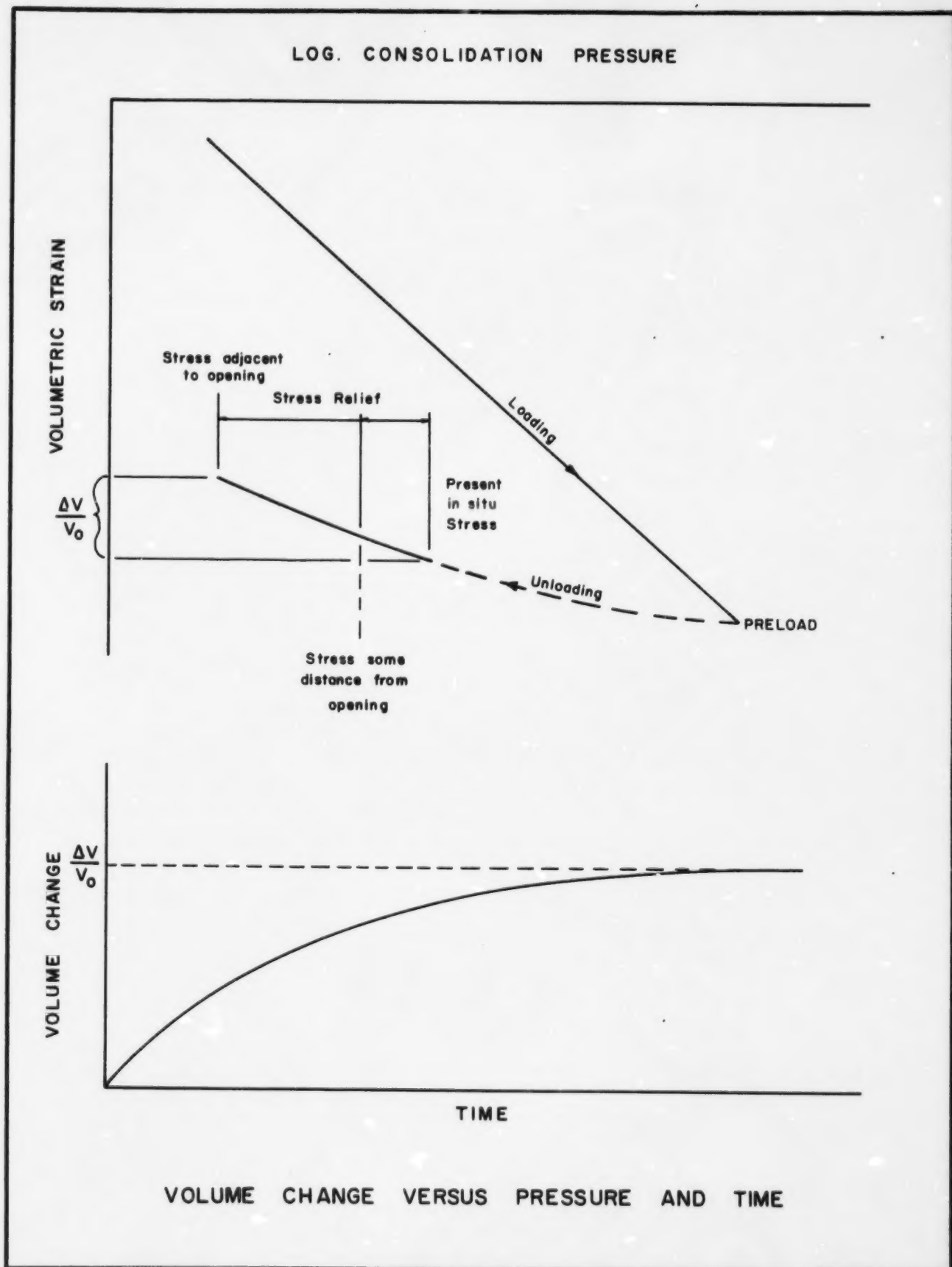


Table 5

COMPRESSION AND SWELL OF NATURAL SOILS

Soil	w_L	w_p	Virgin Compress. Index	Swell Index		Reference
				1000 to 100 kN/m ²	100 to 10 kN/m ²	
Expansive Soil A	84	48	—	0.14	0.25	Dawson, 1957
Expansive Soil B	87	42	0.21	0.05	0.15	Dawson, 1957
Extruded Clay Sample	47	26	0.32	0.10	0.10	Dawson, 1957
Boston Blue Clay Undisturbed	41	20	0.35	0.07	0.09	Mitchell, 1956
Boston Blue Clay Remolded	41	20	0.21	0.07	0.07	Mitchell, 1956
Fore River Clay Undisturbed	49	21	0.36	0.09	0.09	Mitchell, 1956
Fore River Clay Remolded	49	21	0.25	0.04	0.04	Mitchell, 1956
Chicago Clay Undisturbed	58	21	0.42	0.07	0.12	Mitchell, 1956
Chicago Clay Remolded	58	21	0.22	0.07	0.09	Mitchell, 1956
Louisiana Clay Undisturbed	74	26	0.33	0.05	0.08	Mitchell, 1956
Louisiana Clay Remolded	74	26	0.29	0.04	0.07	Mitchell, 1956
New Orleans Clay Undisturbed	79	26	0.29	0.04	0.08	Mitchell, 1956
New Orleans Clay Remolded	79	26	0.26	0.04	0.09	Mitchell, 1956
Montana Clay	58	28	0.21	0.04	0.07	Lambe-Martin, 1957
Fort Union Clay	89	20	0.26	0.04	—	Smith-Redlinger, 1953
Beauharnois Clay	56	22	0.55	0.01	0.04	Mitchell, 1956
Cincinnati Clay	30	12	0.17	0.02	0.03	Mitchell, 1956
St. Lawrence Clay	55	22	0.84	0.04	0.08	Mitchell, 1956
Siburua Clay	70	26	0.21	0.08	0.12	Mitchell, 1956

after Lambe and Whitman, 1969

According to Terzaghi (1943), the rate depends upon the permeability of the rock mass, which in turn has to be considered in terms of permeability of the intact shale, and secondary permeability, or hydraulic conductivity, of the mass. Taylor and Spears (1970) point to structural features such as bedding plane laminations, joints, shear planes, root holes in seat earth, etc., which provide pathways for easier flow of water, and hence are significant for the hydraulic conductivity and rate of swelling.

The magnitude of stress relief can be estimated from mathematical analyses which provide solutions for the stresses around openings of various shapes (Obert and Duvall, 1967), and an estimate of the in-situ stresses prior to mining.

4.2.3 Swelling Clays in Engineering Practice

Geotechnical engineering: In geotechnical engineering, swelling clays and clay shales are recognized for their tendency to cause heave of highly loaded structures, with the heave of spillways constructed in clay shale cuts being a notable example (Morgenstern 1979). Softening due to stress relief and water uptake has also been widely experienced historically, leading to a variety of construction procedures.

Volume changes due to swelling are commonly measured by direct methods such as one-dimensional compression and swelling tests carried out on undisturbed samples. The swelling index, C_s , is obtained from these tests. Typical values are given in Tables 3 to 5. The swelling potential is used for compacted soils; it is defined as the percentage volume change occurring for a sample compacted at optimum moisture content using standard procedures, and allowed to swell under a surcharge of 7 kPa.



It is common in geotechnical engineering to seek empirical relationships between behavioural characteristics such as swelling, and simple index tests such as Atterberg limits and grain size distribution. Table 6 shows an example of a correlation between swelling potential and plasticity index. Further discussion is given by Mitchell (1976) and Lambe and Whitman (1969).

The relationships between strength and the swelling process are part of a larger picture which relates consolidation stress history, weathering, stress changes during loading, pore water pressures during loading and other factors which are beyond the scope of this study. However, the essential features pertinent to this study is reliance upon direct measurement of strength after the clay has been permitted to swell rather than indirect correlations.

Another characteristic closely associated with swelling is disintegration when subjected to wetting and drying cycles. Slaking and dispersion are associated with the disintegration. Although this behaviour occurs in all clays, it is of greater interest to geotechnical engineers in the case of clay shales (Morgenstern, 1979).

Oil-well engineering: In oil-well engineering, shale formations are recognized for their tendency to be unstable and to hydrate and to disperse in the presence of water. Brittle or sloughing shales are one class of instability problem which is due to mechanical and hydraulic fracturing; however, in the context of this report it is the hydratable or swelling shales which are of interest. When drilling with water or fresh water mud, these shales hydrate and become entrained in the mud, adding to the viscosity and creating problems of bit balling, mud rings, plugged flow lines, plugged shale shakers and high solids content. The ensuing enlargement of the wall bore promotes caving of the overlying formation (Technifluids).



Table 6 Approximate Relationship Between Total Expansion, Swelling Potential, and Plasticity Index

Plasticity Index (%)	Swelling Potential ^a (%)	Total Expansion ^b (%)
10	0.4- 1.5	4.5-10.0
20	2.2- 3.8	13.5-18.7
30	5.7-12.2	21.4-18.0
40	11.8-25.0	28.0-35.0
50	20.1-42.6	33.0-40.0

^a For samples compacted at optimum water content using the standard AASHO procedure and allowed to swell under a surcharge of 1 psi.

^b Air dry to saturation under 1 psi surcharge. Seed, Woodward, and Lundgren (1962).

Hydration is defined in the oil-well context as the process of reabsorption of water which was expelled from the clay shale due to high stresses during the compaction period. This process takes place as a result of the stress relief adjacent to the borehole.

Once a shale has swelled by hydration the particles are less tightly bound. Those on the surface slake off, aided by hydraulic or mechanical erosion in some cases. This is known as dispersion. Amounts of swelling and dispersion are often proportional, but not necessarily (O'Brien and Chenevert 1973).

Coal mining: In coal mining, mudstones, shales and seat earths have been identified as subject to disintegration or weathering. Artler (1974) describes a fireclay below the coal and a clay shale layer above it which weather - become soft and disintegrate - when exposed to moist air, especially in summer when the air is also warm. Taylor and Spears (1970) describe strata breakdown by uptake of water, aided by the laminations, bedding planes, shear planes and other structural features which permit ingress of water and permit swelling to be concentrated in mixed-layer clays. The oxidation of pyrite, accompanied by swelling, is also cited by the above authors as being a contributing factor in breakdown.

A study of the Illinois Basin, by Rockaway (1977) describes briefly the cyclothem, and the environment in which "under clays" (seat earth, seat rock, ganister, fireclay) are deposited. It is hypothesized that the underclay was laid down in shallow fresh water, such as a delta. It of course supported the plant growth which became the coal. Typically it is composed of clay minerals, is slickensided, irregularly fractured, and contains traces of plant roots (Huddle and Patterson 1961). Clay mineralogy is quite varied. Thicknesses vary, usually being 1 to 2 m, but occasionally up to 10 m. The lower boundary is gradational with the underlying shale, limestone or sandstone. They are described as lacking bedding but having a vertical zonation: the upper



zone being carbonaceous, and leached of calcium and iron, seldom more than 25 cm thick; the next being more highly plastic and free of carbonates; the lower being generally less plastic, enriched in iron compounds and carbonates, with limestone or iron carbonate nodules and pyrite often being found.

Fractures, fissures and slickensides are the main structural features. Rockaway points out that these directly affect the bearing capacity of underclays below pillars by mechanically weakening the mass.

The studies reported by Rockaway show that the characteristics of the underclay vary greatly both horizontally and vertically, and he concludes that "...it is evident that significant changes in thickness, lithology and induration take place over short distances..." The significance of this observation is that data obtained in one specific location could be inadequate in other nearby areas of the same mine.

The moisture adsorption and swelling behaviours of a coal mine shale are described by Chugh, et al (1980). A 75% illite/mixed layers, 25% kaolinite clay was tested in different conditions of humidity. Pertinent results shown in Table 7 demonstrate a substantial gain in moisture content of 4.1% and four-fold loss in strength as the ambient conditions change from the natural state to full immersion in water. This represents quantitative verification of the observations made by Artler. The report also shows that moisture adsorption occurred over relatively short distances from the free surface (ca. 10 mm); however, the authors feel this is sufficient to cause real swelling problems because of moisture migration into the rock mass along fractures.



Table 7 Summary of Results on Physical Properties of Shale Specimens Under Different Moisture Environments

Physical Property	Natural Moisture Content	90% Relative Humidity	95% Relative Humidity	100% Relative Humidity	Immersed in Water
Unconfined Compressive Strength (C_0) (kg/cm^2)	362.01 (1.64)	287.91 (2.48)	239.11 (1.34)	114.60 (3.8)	96.06 (1.34)
Young's Modulus of Elasticity ($E_1 \times 10^3$) (kg/cm^2)	28.97 (1.53)	26.72 (1.09)	18.87 (0.84)	5.71 (0.22)	3.70 (0.22)
Poisson's Ratio (μ)	0.11 (0.0055)	0.25 (0.0141)	0.44 (0.0214)	0.48 (0.0)	0.48 (0.0)
Water Content (Percent)	1.88 (0.17)	2.31 (0.288)	2.63 (0.108)	4.38 (0.055)	8.00 (0.408)
Maximum Swelling Strain (Percent)		0.157 (0.011)	0.183 (0.013)	0.477 (0.026)	2.520 (0.198)
Induced Stress (kg/cm^2)		41.95 (0.818)	30.87 (0.818)	27.24 (0.420)	92.50 (0.810)
Average Micro-hardness (Vickers Hardness Number)	37.70		35.00 73.5*	33.05	
Average Diffusion Coefficient ($D \times 10^{-10}$) (m^2/s)		2.50 (0.041)	3.45 (0.318)	3.53 (0.088)	40.58 (0.240)

*After air-drying the surface for 32 days
†Standard deviation based on three samples

After Chugh, et al 1980

4.3 Effects of Swelling

The effects of swelling already referred to above fall into three categories:

- volume increase (ie squeezing of well bores and tunnel walls)
- dispersion and sloughing
- strength degradation

A corollary to swelling is swelling pressure, which develops where swelling is physically restrained but where chemically-induced swelling processes are acting.

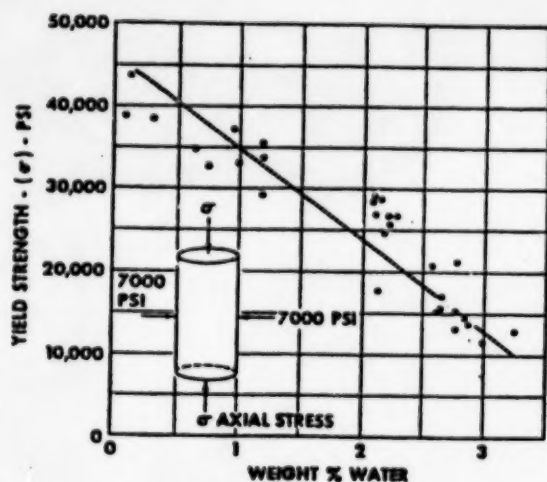
Examples of volume increase have been given in the preceding Sections.

Dispersion and sloughing are commonly cited in the literature, but specific examples have not been found in the present study.

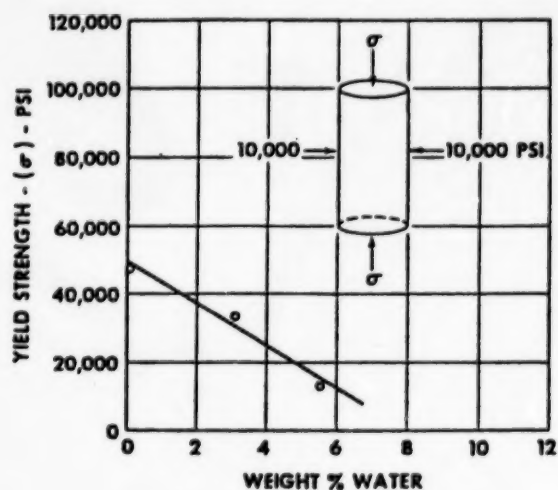
Examples of loss of strength due to adsorption of water for a selection of clay shales are shown in Figure 14 (after Chenevert 1970). Properties of the shales are shown in Table 8. In all cases, uptake of a few percent of water caused substantial strength reductions. Corresponding data on water adsorption are illustrated by Table 9.

Swelling pressures in clays can be very large. Chenevert shows an example where the pressure required to prevent swelling reached 35 MPa in an illite clay shale (Fig. 15).

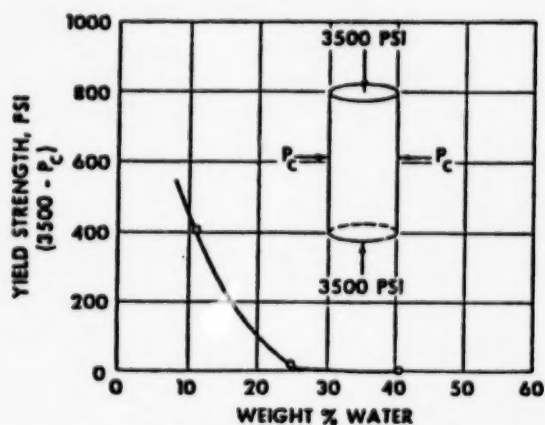




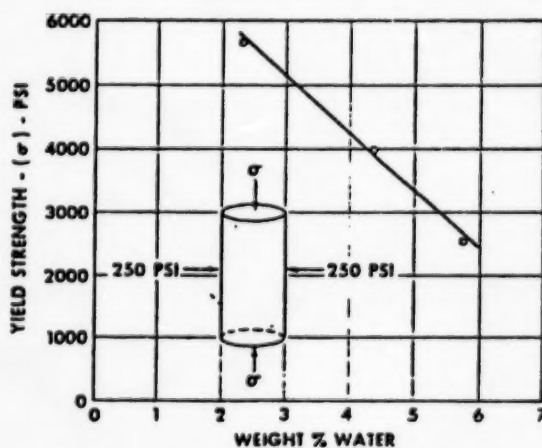
Yield strength of Shale A as a function of water adsorbed.



Yield strength of Shale E as a function of water adsorbed.



Effects of differential pressure and adsorption on yield strength of Shale B.



Yield strength of Shale F as a function of water adsorbed.

YIELD STRENGTH VS WATER CONTENT OF VARIOUS SHALES

After Chenevert (1970)

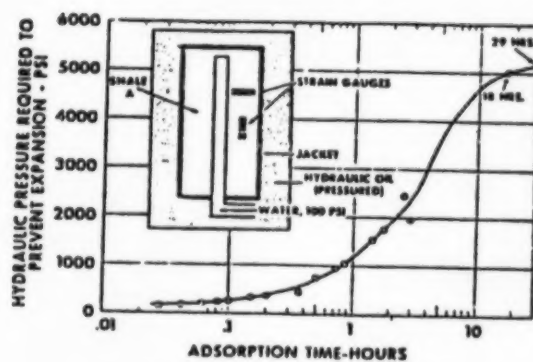
TABLE 8—SHALE PROPERTIES

Shale	X-Ray Analysis (percent)	Weight Percent Water at	Location	Depth (ft)
		a. 0.5		
A	α —Quartz 55.6	1.1	SW Texas	13,000
	Feldspar 20.4			
	Calcite 1.3			
	Dolomite 1.5			
	Illite 14.8			
	Chlorite 4.8			
	α —Quartz 1.5			
	Calcite 0.1			
B	α —Quartz 15.4	4.6	Gulf of Mexico	2,500
	Feldspar 1.2			
	Dolomite 0.5			
	Illite 37.8			
	Montmorillonite 35.1			
	Kaolinite 10.0			
C	α —Quartz 3.2	5.8	Pacific Coast	4,500
	Feldspar 9.0			
	Calcite 0.4			
	Montmorillonite 87.4			
D	α —Quartz 39.3	3.5	Louisiana	16,000
	Feldspar 1.2			
	Illite 46.6			
	Chlorite 7.5			
	Kaolinite 5.4			
E	α —Quartz 61.1	2.4	East Texas	7,800
	Feldspar 16.2			
	Illite 16.0			
	Chlorite 6.4			
F	α —Quartz 0.3	0.8	Central U. S.	300
	α —Quartz 59.6			
	Feldspar 13.2			
	Illite 16.8			
	Chlorite 10.4			

TABLE 9—SORPTIVE PROPERTIES OF SHALES

Shale	Water is:	Temp. (°F)	Weight Percent Water in Equilibrium With a Relative Humidity of: (percent)						
			10	30	50	70	90	95	98
A	Adsorbed	75	0.1	0.6	1.1	1.6	2.6	3.0	3.8
	Desorbed	75	0.1	0.8	1.5	2.2	2.9	3.2	4.2
B	Adsorbed	75	1.0	2.2	4.6	8.7	32.0	40	—
	Desorbed	75	1.7	3.4	5.8	8.8	13.5	15.5	—
C	Adsorbed	75	2.7	4.5	7.7	11.4	16.7	19.0	—
	Desorbed	75	1.5	2.5	3.5	4.7	—	—	—
D	Adsorbed	75	1.6	2.6	3.7	5.0	—	—	—
	Desorbed	75	1.2	1.9	2.4	3.0	4.8	—	—
E	Adsorbed	75	0.1	0.3	0.8	1.5	3.1	4.3	6.8
	Desorbed	75	0.1	0.4	0.9	1.8	4.0	5.8	—
F	Adsorbed	122	0.2	0.6	1.3	2.2	4.4	—	—
	Desorbed	210	0.2	0.6	1.3	2.2	4.4	—	—

After Chenevert (1970)



Hydrational stress developed by adsorption, Shale A.

After Chenevert (1970)

5.0 CHEMICAL STABILIZATION

5.1 General Review

In both oil well and civil engineering practice, soil and rock formations are treated in situ to improve their characteristics. Treatments are generally divided as follows: (i) additives which produce a cement or gel in the voids (such as cement grout), (ii) additives which react with the soil particles to improve strength, reduce hydration and swelling, reduce dispersion of particles, etc.

The treatment methods in the first category, which produce cements or gels in the voids, are not considered further in this report for the following reasons:

- Some of these agents are introduced by grouting methods. Penetration of pumped grouts is limited by the grout pressure that can be used and for underground applications seems entirely inappropriate.
- Other methods require intimate mixing of stabilizing agent with the soil by mechanical means. This is most common in highway and airfield construction. This again seems entirely inappropriate for the purpose of this study.

In the second category, the treatment methods all, in one way or another, rely upon the surface chemistry of the clay fraction of the soil.

The use of chemicals to stabilize problem shales in oil-well drilling and production is widely reported in the literature. A wide variety of materials are used, depending on local availability and other factors.

To a much lesser extent, chemical clay stabilization has been used in civil engineering.

Clay soil stabilization with lime (Ca(OH)_2) has been described frequently in the literature, in connection with highway, rail and airport construction to improve roadbeds and bearing layers. The principle is the replacement of exchangeable cations (usually sodium) by calcium, resulting in flocculation, and increase in strength.



Wagener et al (1981) describe the use of gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) to stabilize dispersive clay which occurs in the floor and embankment of a reservoir in South Africa. The principle of the application was to replace the highly exchangeable sodium percentage which produces loose bonding between clay platelets with calcium, which will bond the platelets strongly and prevent dispersion. The gypsum was introduced by direct application and in solution in the reservoir water.

Sydansk (1983) reports the use of potassium hydroxide to stabilize clays. In laboratory tests, treatment effectiveness was found to depend on concentration, formation temperature and contact time. A 15% by weight aqueous solution of KOH and 16 hours contact time was found to "permanently alter the clay chemistry and render the clay insensitive to fresh water.

Hydraulic fracturing of impervious formations employing a combination of gels and other compounds, and KCl to stabilize the clays, is a well established technique White and Daniel 1980; Chamberlain and Mitchell 1983; Hood and Powell 1982; Schlottman et al 1981). KCl has also been used to stabilize shale intervals by cleaning drill holes using KCl concentrations of 3 to 5% by weight in water (Dees and Spradlin 1982).

Lauzon (1982) notes that the best chemical treatment not only inhibits swelling of water-sensitive shales but must also adsorb quickly and prevent dispersion of fines. Chemicals that inhibit swelling most readily will show charge neutralization in dilute zeta potential experiments.

Thompson et al (1981) describe Cla-Ban, a proprietary polymer additive. Williams and Underdown (1980) discuss new polymers for permanent clay stabilization. Laboratory studies of Polyamines (PA), polyquaternary amines (PQA) and polyacrylamides show the first two provide the best stabilization. As little as 2% PA or PQA is effective in maintaining permeability gained after acidation.



Roehl and Hackett (1982) describe laboratory techniques for testing shale-swelling inhibitors. They report polyacrylamides had little effect on a smectite-kaolinite shale.

Minton (1981) discusses drilling fluids used in drilling North Sea Tertiary shales. A sea water/KCl/polyacrylamide mixture is used for "hydratable" smectite and illite clays.

Hill (1982) reports that cation exchange capacity of the clay is useful for predicting the concentrations of clay control additives. The CEC can be easily determined by adsorption of methylene blue and by sodium ion exchange with ammonium acetate.

Thomas and Crowe (1978) refer to use of fluoboric acid to reduce CEC and hence enhance stability. This treatment has not been investigated for the purposes of this report, but could be reviewed in follow-up studies.

Penkov and Levik (1980) report successful clay stabilization with gypsum-potassium muds with $\text{pH} < 8.5-9.5$, 1000-2000 mg Ca^{++} and 5000-15000 mg K^+ /litre.

Drilling fluids consisting of oil and oil water emulsions (oil-base muds) are reportedly effective. However, applications underground with oil or oil-water mixtures do not appear attractive and have not been pursued in this report.

Zeinalov et al (1981) also describe gypsum mud, with a recommended Ca concentration of 900-1100 mg/l.

McLauchlin (1976) describes a class of agents known as surfactants which impart oil wetness to the clay surface so that water will not wet it. This treatment will, in principle, deal effectively with humid air or dust-laying water; however, it will have no effect on swelling by uptake of formation water. Organic amine surfactants act as illustrated in Figures 16 and 17. The long chain molecule is attached by nitrogen lodged in the cation exchange sites of the particle or layer surface.



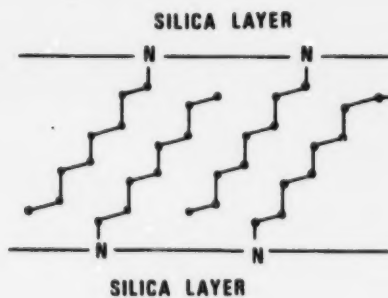


Fig. 16 - Cationic surfactant adsorbed between clay sheets.

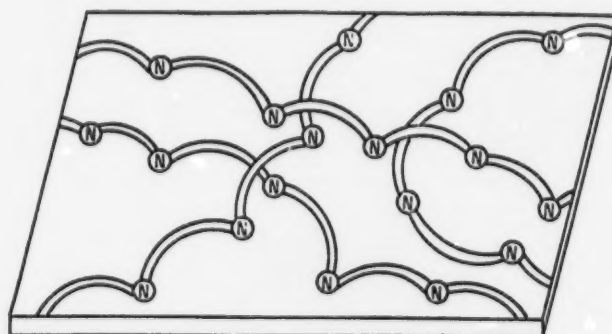


Fig. 17 - An organic polymer adsorbed on a clay surface

after McLauchlin

What emerges from these reports is that a small variety of water-based chemical additives have been used repeatedly, with success. These are potassium salts, inorganic polymers, and organic polymers. Other chemicals are, however, in use. A comprehensive listing of products commercially available in Canada is given in Table 10.

5.2 Potassium

As a dissociated cation in an electrolyte, potassium will displace the sodium or calcium on the clay surface and because of its extremely low hydrational energy and high bonding energy will initiate dehydration of the platelets. The potassium ion causes the clay plates to be pulled closer and closer together meanwhile forcing out the attached water molecules. The diameter of the potassium ion being small allows the contraction to continue until the clay lattices and layers reach minimal dimensions.

O'Brien and Chenevert (1973) describes how potassium ions are of a favourable size and low hydrational energy which enables them to replace exchangeable cations, promoting interlayer dehydration and a high degree of fixation. Testing is recommended to determine effectiveness of inhibitive fluids. As a guide, KCl concentration in water at 3 to 5% seems to cause the greatest improvement. To allow for base exchange reactions, they recommend operating with 5% for illites and 10% for montmorillonites.

KOH may be used for alkalinity control. Maintaining pH levels in the 10-12 range will reduce the level of corrosion. Foaming can be a problem with KCl concentrations above 5%. A defoamer, such as IMCO "Defoam-L" should be used. Potassium is available as non-dispersed KCl, dispersed KCl, KOH-lignite and KOH-lignosulphonate. Non-dispersed KCl is seldom used at concentrations above 4%. KOH is desirable when high Cl-ion concentration is objectionable.

Potassium test strips are used much like litmus paper and will measure concentrations between 300 and 1000 ppm. They are easy to use and relatively accurate.



Table 10 - Shale Control Inhibitors

PRODUCT TRADE NAMES	PRODUCT DESCRIPTION (Generic, Chemical or Other)	SYSTEMS USED IN				DISTRIBUTED IN CANADA BY
		WATER BASE			Fresh Water	
		NONDISPERSED DISPERSED	INHIBITED	BRACKISH		
AMMONIUM SULPHATE	Ammonium sulphate		•	•		MOST
BARAFLOC B	Organic polymeric hydrolytic powder		•	•		BAROID
CALCIUM CHLORIDE	Calcium chloride		•	•		MOST
CROSFLOC CFA-70	Polycrylamide P/B	•	•	•		HART CHEMICAL
DAP	Diammonium phosphate	•	•	•		MOST
DETERGENT	Detergent	•	•	•		MOST
ENKAPULATOR	Polycrylamide		•			UNITED MUD
FL-1	Improved organic polymer	•	•	•		MOST
4M + 4	Hydrolyzed polyacryl amide	•	•	•		MAGCOBAR
GYPSUM	Calcium sulphate		•	•		MOST
H-S POLYMER	Polycrylamide		•	•		BAROID
IMCO MOLE COAT	Asphaltic solids	•	•	•		IMCO SERVICES
IMCO IE PAC	Caulitized potassium lignite	•	•	•		IMCO SERVICES
IPI-WD	High softening point asphalt		•	•		MILCHEM
MIL-SOS	Hydrolyzed polyacrylamide	•	•	•		MILCHEM
PLASTER	Calcium sulphate		•	•		ALL
POLYGUARD	Shale inhibitor	•	•	•		UNITED MUD
POLY PLASTIC	Shale stabilizer	•	•	•		I.D.F.
POLY S	Shale stabilizer	•	•	•		CANAMARA SUPPLY
POTASH	Potassium chloride (KCL)	•	•	•		MOST
RM-88	Polymer	•	•	•		ALL
SHALE-GARD	Water dispersible asphalt	•	•	•		MILCHEM
SHERIDALE	Asphaltic shale inhibitor	•	•	•		CANAMARA SUPPLY
SS-100	Shale encapsulator		•	•		I.D.F.
SST 202	Aluminum salt	•	•	•		MILCHEM
STABILHOLE	Brown asphalt	•	•	•		MAGCOBAR
SULPHATE OF POTASH	K ₂ SO ₄	•	•	•		MOST
SUPER LIME FLOW	Gypsum	•	•	•		MOST
TARGUARD	Core protective	•	•			I.D.F.
X-PEL-G	Treated gilsonite	•	•	•		MOST

From Oilweek

5.3 Polymers

McLaughlin et al (1976) note that sodium clays are the important form of swelling or dispersing clays. A successful method of treatment is replacement of the Na^+ by K^+ , Ca^{++} or NH_4^+ ions. The sodium ion hydrates to many times its size, while the others do not; thus there is less swelling. However, this is temporary protection in oil field production, since sodium ion from the formation water replaces the ion induced in the drilling fluid fairly quickly. Therefore, for long term stability during hydrocarbon production, they advocate polymers.

A new series of polymer materials has proved to be effective in treating water sensitive formations. These polymers adsorb tenaciously to surfaces of clay minerals and other formation fines, via multiple ionic attachments, helping to prevent clays from swelling and fines from migrating should foreign water be introduced into the formation. The polymers win easily in competition with other simple ions (such as ammonium, calcium or potassium) for being deposited on clay surfaces. Furthermore, once deposited on clay surfaces, the polymers are not easily replaced by exchange with other ions. Subsequent acidizing treatments cause no detectable harm to the ability of the polymers to protect the clay.

5.4 Inorganic Polymers

Inorganic polymers such as hydroxyl aluminium and zirconium oxychloride provide stable treatment materials. Many cations are united in a complex structure in the polymer, and these fill many cation exchange sites on the particle or layer surface. The resulting product is difficult to dislodge. Mildly acidic conditions (pH 4 to 5) are necessary to keep the chemical in solution, and acid treatment of the clay can remove these polymers. pH control of mine water is therefore indicated, which may not be possible in circumstances where carbonates in the stratum may interfere with the reactions. (McLaughlin, et al 1976).



Peters and Stout (1977) report successful results with zirconium salts, which in water produce hydrolyzed polynuclear ions that perform as ions of much higher charge.

Coppel et al (1973) describe results from wells treated with hydroxy-aluminum, which they refer to as slightly acidic, non hazardous, colorless, odorless, non toxic, and requiring no unusual handling procedures. Laboratory evidence is that polymerization on the clay mineral surfaces takes place rapidly during the first few hours, and becomes slower after about 12 hours.

Both the zirconium and hydrolyzed aluminum were used in the cases reported by Peters, and Coppel et al, to control dispersion and maintain or improve well production rates. Although swelling and dispersion are often described as two manifestations of the same clay-cationic reactions, it may be that these chemicals are less effective in swelling control than in preventing dispersion.

5.5 Organic Polymers

Certain organic polymers (COP) have been found to be effective and long lasting for treating exposed surfaces of clays. They are characterized by multiple nuclei or groups which can react with cation exchange sites on the particle or layer surfaces. The result, as illustrated in Figure 17 is a connected assembly of ionic nuclei (rather than cations) linked together like a chain link fence.

Organic polymers are not acidic nor do they require an acidic pH whilst in solution. Carbonate components in the clay have no effect. The organic polymers can be used with solutions of NH_4Cl , KCl , CaCl_2 and HCl , all of which are themselves used as clay stabilizing agents. (McLaughlin et al 1976)

The organic polymer can be mixed by simply stirring it into clear water. Quantity and concentration have to be determined for each given clay to be treated.



5.6 Potential Applications in Coal Mines

The foregoing review has pointed to two swelling mechanisms:

- swelling due to stress relief (rebound)
- swelling due to ion exchange

In the underground mine situation, two possibilities can then be identified:

- rebound, with uptake of formation water and no ion exchange phenomena.
- rebound, plus swelling due to ion exchange effects because of the presence of fresh dust-layering water or humid air.

It has been shown that the categories of treatment potentially applicable to mining are:

- polymers which seal the surface to which they are applied, thus limiting swelling to that associated with uptake of formation water.
- cation treatments which prevent or inhibit ion exchange-related swelling, thus limiting swelling to that associated with stress relief : the water may be either formation water or mine water.

It would seem that rebound is difficult to prevent because stress relief and formation water are largely unavoidable; however, there is a possibility that the right kind of cation used as a treatment could actually prevent rebound (and potentially strengthen the clay shale).

The principal questions to be addressed in using chemical treatments in coal mines are:

1. What is the goal of the treatment - to stabilize in situ material, to improve it or to minimize degradation?



2. What chemical treatments would provide the best results?
3. How much chemical should be used and how can it be applied?
4. What are the environmental consequences?
5. How permanent is the treatment?

These questions cannot be answered from the literature and other sources of information known to us at this time, and so a program of investigation is needed. Part of this investigation would be a laboratory testing program, as described in the next section. However, laboratory tests should be done in consort with field studies. Although recommendations for field studies are not within the scope of this study, it is clear that the underground moisture environment and details of the softening process, to name only two questions, need clarification, and that in situ studies should receive a high priority.

5.7 Toxicity and Costs

Application of the shale stabilizers in the mine environment will expose the miners to any hazardous characteristics of the chemicals which must also be considered as criteria in the selection process.

The safety characteristics of most of the shale stabilizers appearing in Table 10 are summarized in Table 11. Only two of the products are classified as hazardous and most are general food additives. It is recommended, however, that the Material Safety Data Sheets be requested when purchasing a specific chemical.

The cost of oil well chemicals is dependent upon availability, demand, packaging, format and size of the purchase. Most suppliers are reluctant to quote price verbally; stating that prices are negotiable. One supplier did give prices for several products, however, stated that these may be variable with a firm order.



<u>Product</u>	<u>Unit Size</u>	<u>Unit Cost*</u>
4 M+L	20 liters (liquid)	74.86
Alcomer 120L	25 kg (liquid)	95.77
Stabilhole	20 lb sack	26.93

*FOB Walton, Nova Scotia



TABLE 11 - SHALE STABILIZERS SAFETY DATA

PRODUCT/ SUPPLIER	CHEMISTRY	HAZARDOUS INGREDIENTS	HAZARDOUS DECOMPOSITION PRODUCTS	HEALTH HAZARD DATA	FIRE AND EXPLOSION DATA	SPECIAL PRECAUTIONS	INCOMPATIBILITY	HAZARD CLASS	COMMENTS
Calcium Chloride NL Baroid	<chem>CaCl2</chem>	None	None	Eye and skin irritation	Non combustible	Avoid eye and prolonged skin contact	Corrodes metal	Not hazardous	A food additive
Gypsum NL Baroid		None	None	Nuisance dust	Non combustible and explosive	None	None	Not hazardous	
EZ Mud NL Baroid	Polyacrylamide Polyacrylate	None	None	May cause eye & skin irritation	Flash point > 200° F Extinguish with chemical foam	Do not take internally Avoid skin & eye contact Causes slippery floors	None	Not hazardous	A food additive
Potash NL Bariod	<chem>KCl</chem>	None	None	Minor skin, inhalation, gastro- intestinal, severe eye irritant	Non combustible or explosive; does not support combustion	Avoid prolonged eye and skin contact	None	Not hazardous	
Stabil Hole Dresser- Mascobar	Naturally occurring asphalt	None	<chem>CO2</chem> , <chem>CO</chem>	Fumes, dust may irritate eyes & upper respiratory tract, may fog, <chem>CO</chem> , photosensitize dry chemical skin	Flash point > 700° F extinguish with water, fog, <chem>CO</chem> , photosensitize dry chemical skin	Avoid prolonged eye and skin contact	None	Not hazardous	
Percol 156 Dresser- Mascobar	Anionic Polyacrylamide	None	None	Slight skin and eye irritant	Flash point > 100°C will support combustion	Avoid prolonged eye and skin contact	Cationic polymers	Not hazardous	A food additive

TABLE 11 (Continued)

PRODUCT/ SUPPLIER	CHEMISTRY	HAZARDOUS INGREDIENTS	HAZARDOUS DECOMPOSITION PRODUCTS	HEALTH HAZARD DATA	FIRE AND EXPLOSION DATA	SPECIAL PRECAUTIONS	INCOMPATIBILITY	HAZARD CLASS	COMMENTS
Classta II Haliburton	N/A	None	Toxic gases produced by incomplete thermal decomposition	Skin and eye irritant	Extinguish with foam, dry chemical, CO ₂ water spray	Avoid eye and skin contact	Strong alkaline materials	Not hazardous	
Potassium Sulphate (most)	K ₂ SO ₄	N/A	N/A	Moderate to highly irritating	N/A	N/A	N/A	Dangerous	A food additive
Ammonium Sulphate (most)	(NH ₄) ₂ SO ₄	N/A	N/A	Slightly irritating	N/A	N/A	N/A	Dangerous	A food additive

Source: Material Safety Data Sheets

6. PROPOSALS FOR LABORATORY TEST PROGRAMS

6.1 Study of the Seatearths

A program of systematic sampling of the seatearths for laboratory determination of properties is recommended. This will provide base data on the nature and distribution of materials which may require treatment. Laboratory procedures are as follows:

- "Clay" mineralogy:untreated materials
 - X-ray diffraction
 - Differential thermal analysis
 - Cation exchange capacity
 - Exchangeable sodium percentage
- Physical properties:untreated materials
 - water content
 - *- bulk density
 - *- grain size distribution
 - permeabilty
 - *- Atterberg limits
 - *- porosity
 - *- dispersion
 - slake durability
 - adsorptive isotherm
 - swelling index
 - swelling strain
 - undrained shear strength

It is recommended that all samples be subjected to the tests marked with an asterisk*. This will permit a classification system to be developed. Representative samples of each class should then be subjected to the full battery of tests, to get some feel for the problem.

It is acknowledged that it is possible to proceed directly to trial chemical treatment methods, without acquiring this base data, and such an approach could be completely satisfactory, for a while. However, it is our opinion that the use of such an approach means constantly experimenting with treatments and perhaps never acquiring the predictive capability that a more fundamental approach gives.



6.2 Tests to Determine Effect of Chemical Stability Agents

This round of tests would involve the following sets:

- different chemicals
- application rates
- method of application, including secondary requirements such as pH control

The results would be measured by means of:

- dispersion tests
- swelling strain tests
- swelling pressure tests
- unconfined compression tests (or indirect methods such as point load index)

Further literature review is a necessary part of this testing program. The literature generated by our review suggested overall results by various researchers however, was not definitive in terms of detailed laboratory test procedures. Direct contact should be established with researchers such as O'Brien, Chenevert, McLaughlin, and Coppel to obtain more detail on testing procedures.

The literature survey indicates that potassium is the primary form of cation treatment, with calcium a somewhat distant second, although calcium in the form of lime is quite clearly cheap and non-toxic. These are obvious first choices for chemicals to test. In the polymer field, proprietary products such as Haliburton's Sta-Cla and Technifluids's SS-100 polyacrylamide are candidates. The other materials referred to have not been described in the literature we have reviewed, and some further inquiries are called for.

The proposed laboratory studies would not give absolute results in the sense that they would show whether a particular treatment will prevent problems in the mine. They are, however, realistically comparative tests, so that what works best in the laboratory will, in all probability, work best in the mine. As such, they are of real value.



7. CONCLUDING STATEMENT

In the course of this study the authors have become convinced that the idea of developing chemical agents for stabilization of coal measures strata is sound and worthy of pursuit, and that the experience of the oil industry provides an excellent beginning. In accordance with the terms of the study, we have addressed the laboratory programs which might be considered to achieve the goal. However, we have also been made aware that knowledge of the conditions in the mines is of great importance. For example, the underground moisture environment and the details of the softening process need clarification. Consequently, we feel that programs of laboratory tests should be done in consort with field studies, and that this would be much more productive than just laboratory studies. In fact, it would be difficult to be supportive of further work based on laboratory studies alone.



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APPENDIX 1

03SQ.23440-3-9034

STATEMENT OF WORK

1. Through correspondence, telephone conversations and personal visits (with prior approval of the Scientific Authority) become familiar with the problem of soft floor strata in coal mines, the mechanism of deterioration of coal measures strata by water and the requirements expected of a successful chemical additive.
2. Through correspondence, telephone conversations and personal visits (with prior approval of the Scientific Authority) survey the range of oil well stabilizing chemicals and assess which of them are most likely to be effective on the basis of the following:
 - a) toxicity
 - b) required concentrations
 - c) effect on strata
 - d) cost, and
 - e) detrimental side effects
3. Having selected the most effective agents on the basis of the items above, design a laboratory test program to determine the efficacy of each of the selected agents and the optimum concentrations required. Consideration should be given to the types of strata to be used, the collection and preparation of samples, the tests to be conducted and the controls to be employed to ensure accuracy.